TECHNICAL NOTES

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

No. 832

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National Bureau of Standards

Washington
November 1941

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SUMMARY

About two-thirds of the residual stress in cold-worked SAE 1050 steel tie rods was relieved by heating 30 minutes at 600° F. Cold-worked austenitic stainless-steel tie rods could be heated at temperatures up to 1000° F without lowering the important physical properties. The corrosion resistance, in laboratory corrosion tests, of straight 18:8 and titanium-treated 18:8 materials appeared to be impaired after heating at temperatures above 800° or 900° F. Columbium-treated and molybdenum-treated 18:8 steel exhibited improved stability over a wide range of temperatures. Tie rods of either material could be heated 30 minutes with safety at any temperature up to 1000° F. At this temperature most of the residual stress would be relieved.

INTRODUCTION

A high percentage of the streamline tie rod failures examined at the National Bureau of Standards have been attributed to torsional fatigue due to synchronous vibrations. One characteristic feature of such failures, in the streamline portion of the tie rod, is that fracture invariably starts at or near the intersection of the minor axis with the surface. A typical fracture of this kind in an 18:8 corrosion-resistant steel tie rod is shown in figure 1.

The reduction to streamline section is usually performed by rolling or drawing. In most tie rods the high physical properties required are produced by cold-working during these operations. Such tie rods naturally contain very high residual (internal) stresses. Residual stresses may be dangerous in highly stressed members, such as tie rods, particularly when the distribution of stress is such

that it acts in the same direction as the superimposed service stress.

In most tie rods, the residual stress is so distributed that the highest tensional stress occurs at the intersection of the minor axis with the streamline surface, which is the point at which the fractures start. It is probable, therefore, that high residual stresses are important contributory causes of failure in these tie rods.

In the attempt to reduce the number of failures of this type, an investigation was undertaken at the request of the Bureau of Aeronautics, Navy Department, to determine whether or not the residual stress could be substantially relieved by a relatively low-temperature heat treatment without materially affecting the physical properties of the material.

MATERIALS

Streamline tie rods of the materials shown in table 1 were included in the investigation.

TABLE 1.- MATERIALS USED IN INVESTIGATION

Material	Size (a)	Chemical composition (percent)							
		C	Cr	Ni	Ti	Cb	Mo	Cu	Al
A (SAE 1050 steel)	3/8-24	[b]0.45-0.50	------	-----	----	----	----	--	----
B -------do-------	3/8-24	[b].45- .50	------	-----	----	----	----	--	----
C (18:8 stainless)	3/8-24	[b]--------	18	8	----	----	----	--	----
D -------do-------	3/8-24	[b]--------	18	8					
E -------do-------	1/2-20	0.12	19.1	9.0					
F -------do-------	10-32	[b]--------	18	8					
G (18:8 + titanium)	10-32	[b]--------	18	8	0.4				
H -------do-------	1/2-20	.05	18.5	8.7	.37				
I -------do-------	5/8-18	[b]--------	18	8	.4				
J (18:8 +columbium)	10-32	[b]--------	18	8	----	0.75			
K -------do-------	1/2-20	.09	17.8	9.7	----	.77			
L -------do-------	5/8-18	[b]--------	18	8	----	.75			
M (18:8 + molybdenum)	5/8-18	0.06-0.07	17.44	10.21	----	----	2.96		
N (18:2 stainless)	10-32	[b]--------	18	2					
O -------do-------	1/2-20	0.10	17.3	2.17					
P -------do-------	5/8-18	[b]--------	18	2					
Q (16:1 stainless)	10-32	[b]--------	16	1	----				
R -------do-------	1/2-20	.11	15.5	1.65					
S -------do-------	5/8-18	[b]--------	16	1	----				
T (K-monel)	10-32	[c]--------	----	66	----	----	----	29	2.75
U -------do-------	1/2-20	[c]--------	----	66				29	2.75
V -------do-------	5/8-18	[c]--------	----	66				29	2.75

[a]Sizes given in Navy specification 49T9a refer to threaded ends.
[b]Nominal composition. [c]Typical analysis of a K-monel alloy.

SCOPE

The scope of the investigation may be outlined as follows:

Measurement of residual stress

Relief of residual stress by heat treatment

The effect of heat treatment on the corrosion resistance of the materials

The effect of heat treatment on the microstructure of the materials.

MEASUREMENT OF RESIDUAL STRESS

The elliptical shape of the streamline tie rods would not permit the use of the most precise method of residual stress determination originated by Howard (reference 1) and Heyn (reference 2), developed and modified by Merica and Woodward (reference 3) and Sachs (4), and used by Green (reference 5) to estimate the residual stress in quenched steel cylinders and by Kempf and Van Horn (reference 6) to investigate the relief of residual stress in aluminum alloys. The split-ring method used by Hatfield and Thirkell (reference 7) or the slit-tube method of Crampton (reference 8) were, of course, not applicable to solid elliptically shaped tie rods. However, the method used was somewhat similar to that of Crampton in that stress was partially relieved on one side of a plane of symmetry and the resultant distortion of the remaining material was measured.

The residual stress distribution in the tie rods as received was determined by measurements on the major and minor axes of the streamline cross section. Stress distribution about an axis parallel to the minor axis was determined by measuring the change in width after partially splitting the tie rod longitudinally by a saw cut. The cut ends of cold-worked tie-rod specimens approached each other, tending to close the saw cut. This indicated initial tension along the longitudinal plane of the minor axis and compression at the ends of the major axis. The partial residual stress along the longitudinal plane of

the minor axis (one-half width of saw cut from center of the major axis) was calculated, as follows:

The deflection caused by partially splitting a section by a saw cut was measured by the change in width at the cut. The radius of curvature was calculated by the formula

$$R = \frac{L^2}{2d}$$

where R radius of curvature, inches

L length of saw cut, inches

d deflection (one-half the change in width)

The partial residual stress was then calculated by the formula

$$S_1 = \frac{EC_1}{R}$$

where S_1 partial residual stress near the center of the major axis, pounds per square inch

E modulus of elasticity (3×10^7 lb/sq. in.)

C_1 distance from saw cut to neutral axis of segment - 0.42 times width of segment

Stress distribution about an axis parallel to the major axis was determined by measuring the deflection after machining specimens on one side to approximately half their original thickness. Partial relief of residual stress due to machining caused the specimens to become convex on the machined side. This indicated that the residual stress was compressive at the major axis and was tensile at the end of the minor axis. The partial residual stress at the end of the minor axis was measured as follows:

The amount of deflection at the end of the minor axis was measured with a micrometer depth gage having a length (chord) of 4 inches. The radius of curvature was calculated by the formula

$$R = \frac{L^2}{2d}$$

where L one-half gage length, inches

d deflection, inches

The partial residual stress was then calculated by the formula

$$S_2 = \frac{EC_2}{R}$$

where S_2 partial residual stress at the end of the minor axis, pounds per square inch

C_2 distance from end of minor axis to neutral axis - 0.58 thickness of specimen after machining, inches

A mean value of residual stress at the ends of the minor axis, therefore, would be the sum of the two partial residual stresses - the partial stress about an axis parallel to the minor axis S_1 and the partial stress at the end of the minor axis S_2 about an axis parallel with the major axis. In most tie rods both stresses were tensile and in some of them the total residual stress was very high. The deformation caused by partial relief of high residual stresses in some of the tie-rod specimens is shown in figure 2.

Examples of the partial residual-stress measurements made for SAE 1050 steel tie-rod specimens (materials A and B) as received are given in tables 2 and 3. Table 4 gives the approximate residual stress (the sum of the two partial residual stresses) at the end of the minor axis, obtained by similar measurements on all materials included in the investigation.

In the calculation of the partial residual stresses, it was assumed that the stress distribution in the plane under consideration was linear. This assumption involved some error, as the actual stress distribution probably was not linear. For this reason the calculated average partial stress in the plane of the minor axis probably is too high, and the calculated partial stress at the end of the minor axis probably too low. The sum of these partial stresses, however, is believed to be a fair index of the actual residual stress at the end of the minor axis.

It is estimated that the experimental error involved in the measurements used in calculating the residual stress

is less than 5 percent. In this connection, it will be noted in table 2 that actual measurements taken at various lengths of cut on specimens of A and B materials gave (except for the first readings on each) values for radius of curvature well within the calculated error.

With all of the materials except 18:2, 16:1 and K-monel, severe cold-working during fabrication was relied upon to produce the high physical properties required in tie rods. It is understood that these materials also received some cold-working during fabrication but were heat-treated afterward to obtain the required physical properties. It is evident from the measurements made on these specimens that the residual stress distribution resulting from heat treatment is just the opposite of that obtained from cold-working. Thus, in all the 18:2 and 16:1 tie rods and in the smallest size K-monel tie rods the stress about a plane parallel to the minor axis was found to be compressive instead of tensile. In the two larger K-monel tie rods the stress was tensile but was very small compared to the values obtained with materials not heat-treated after fabrication. No attempt was made to relieve the relatively small amount of residual stress in these tie rods by further heat treatment.

RELIEF OF RESIDUAL STRESS BY HEAT TREATMENT

It was assumed that, in heating, the residual stress would be relieved equally in all directions. The partial and mean residual stresses would, then, remain in the same ratio throughout the heat treatment. In the tests outlined below, the partial residual stress was determined by splitting the ends of specimens with a saw cut. The residual stress was then calculated by dividing by the ratio of partial to mean residual stress displayed by each material in the "as received" condition. This ratio is given in the last column of table 4.

Specimens of SAE 1050 steel tie-rod materials A and B were heated for periods of 30 minutes and of 2 hours at temperatures between 200° and 900° F. Residual stress measurements for material A are given in table 5. These, together with tensile strength, permanent set, and elongation in 2 inches, obtained on specimens heated 30 minutes at the same temperatures, are shown diagrammatically in figure 3. Relief of residual stress apparently started

almost immediately on heating. The stress fell off abruptly above 200° F, and at 600° F about two-thirds of the stress had been relieved. Heating for a longer period (2 hr) relieved the stress more effectively, especially at the higher temperatures, but could be expected to have a proportional effect on the tensile properties.

The effect of heat treatment (30-min period) on the physical properties of the SAE 1050 steel tie-rod specimens was to cause a marked increase in tensile-strength and permanent-set values up to 400° F and rapid decrease at temperatures above 500° F (fig. 3). Because of the initial increase these properties did not fall below the original values under 600° F, at which temperature most of the residual stress had been relieved.

Examples of residual-stress measurements made on 18:8 corrosion-resistant tie-rod specimens are given in table 6. Curves illustrating the effect of heat treatment on various sizes of tie rods of this material are shown in figure 4. Curves of the same general character were obtained for like sizes of 18:8 materials containing alloy additions of titanium, columbium, and molybdenum. In general, heating at temperatures up to 700° F appeared to have little effect on relief of stress. Stress relief was most rapid at temperatures between 800° and 1000° F. At 1000° F most of the residual stress had been relieved.

It was noted that tie rods of different size varied considerably in regard to uniformity of residual stress in the "as received" condition. In tie rods of intermediate size (1/2-20) the initial stress was much more uniform than in the larger (5/8-18) or smaller (10-32) sizes.

Much of the "scatter" obtained in residual-stress measurements made with specimens heated at low temperatures was probably due to nonuniform initial stress. With size 1/2-20 specimens the scatter was largely eliminated at higher temperatures. The curves showing the heating characteristics of the larger or the smaller size tie rods were smoothed out, to some extent, by plotting the highest values obtained at any given temperature for the three heating periods used. This, in effect, increased the number of specimens in the low-temperature range.

Increasing the time of heating increased the amount

of stress relief at higher temperatures (800° to 1000° F) but apparently had little effect at temperatures below 700° F (fig. 5).

The effect of heat treatment on the tensile properties of specimens of 18:8 (C) material was found to be quite similar to that on columbium-treated (K) material (figs. 6 and 7). Both materials showed a slow and comparatively small increase in strength with heating temperature to a maximum at 800° to 900° F. Above these temperatures the rate of decrease was slow, so that the tensile strength, yield strength, and permanent set were maintained over a considerable range of temperature at values in excess of the original.

With the titanium-treated material (H), the increase in strength with heating was much greater and occurred over a smaller range of temperature (fig. 8). At temperatures above 900° F the rate of decrease was rapid, but, because of the high maximum value attained, the strength at 1000° F was still in excess of the original.

With all three materials, heat treatment lowered the elongation values in the temperature range of maximum tensile strength. With the titanium-treated material especially, the elongation at intermediate temperatures was comparatively low. However, as most of the fractures occurred at or near the edge of grips, the elongation measurements must be regarded as representing minimum values. Moreover, in the temperature range of most interest to the investigation (above 900° F), all three materials showed increasing elongation values. At 1000° F, the elongation values were in all cases equal to or greater than the original.

The values of "yield strength" (offset - 0.2 percent) shown in the figures were determined from the stress-strain curves. It is defined as that stress at which the stress-strain curve is intersected by a line which intercepts the abscissa at 0.002 inch per inch strain and is parallel to the slope of the stress-strain curve at the origin.

The permanent set was determined by measuring the difference in strain at a small initial load after loading and unloading to successively higher loads until sets of about 0.0002 inch per inch were noted. These data were referred to zero stress by plotting to a large scale and drawing a smooth curve through the observed points.

Tensile tests on heat-treated specimens of materials E and M were not made. Vickers indentation tests, however, were made on specimens of these materials after heating 30 minutes at temperatures ranging from 300° to 1800° F. The Brinell numbers of these specimens are shown diagrammatically in figures 9 and 10. These curves display variations of indentation numbers with temperature similar to those exhibited by the tensile strength curves obtained for like materials. It is probable that yield strength, permanent set, and elongation values would also display similar variations with temperature.

The tensile properties of material M as received are given in table 7. This table also contains the tensile properties of specimens of size 5/8-18 tie rods of 18:2, 16:1, and K-monel (P, S, and V) materials as received.

Specimens of materials E, H, K, and M were tested, full size, in the Izod machine after heating 30 minutes at temperatures ranging from 300° to 1400° F. All specimens of E, K, and M materials merely bent over without breaking. Complete breaks were obtained only on specimens of H material heated at 800°, 900°, and 1000° F. Even in these cases the specimens bent considerably before fracture. The tup dragged along the specimen and the values of energy consumption, therefore, had no significance. The tests indicated, however, that heating for 30 minutes at temperatures up to 1400° F did not produce extreme brittleness in any of these materials.

EFFECT OF HEAT TREATMENT ON THE CORROSION RESISTANCE OF THE MATERIALS

The residual stress measurements and tensile tests indicated that most of the residual stress could be relieved by heat treatment without seriously lowering the original mechanical properties of the materials. With the stainless steel tie rods, however, stress relief was found to be most effective at heating temperatures which might be detrimental to the corrosion resistance. It was thought advisable, therefore, to make a number of laboratory corrosion tests on these materials. Particular attention was paid to the stainless steels containing additions of titanium, columbium, and molybdenum to determine whether or not the stabilizing effects of these

elements would permit heating at higher temperatures without impairing the corrosion resistance of the materials.

The effects of heat treatment on the corrosion resistance of materials E, H, K, and M were compared by subjecting heat-treated specimens to the following laboratory corrosion tests:

(a) Salt-spray test

(b) Boiling-nitric-acid test

(c) Boiling-copper-sulphate sulphuric-acid test

(a) Salt-Spray Test

Six-inch specimens of the stainless-steel tie-rod materials (E, H, K, and M) were heated 30 minutes at temperatures ranging from 300° to 1400° F. One end of each specimen was then rounded off and the specimens were polished on abrasive papers finishing with 400 aloxite. After passivation in 20-percent nitric acid at 135° F they were exposed in a vertical position to the spray of a 20-percent solution of sodium chloride. The test was continued for about 1200 hours (1000 hr for material M) with daily inspection of the specimens. The appearance of the four sets of specimens after testing is shown in figures 11, 12, 13, and 14.

At the end of 1200 hours, specimens of E (18:8) material heated at temperatures below 1000° F showed only superficial staining with no evidence of progressive corrosive attack (fig. 11). The superficial stains, when present, were usually traceable to a slight attack at the ends. Specimens heated at 1000° and 1100° F developed rust stains within 24 hours. The corrosive attack then progressed steadily throughout the test. After 1200 hours, the specimens showed a considerable amount of pitting both at the ends and along the sides. The specimen heated at 1200° F showed only slight pitting after 1200 hours while the specimen heated at 1400° F was not attacked.

Specimens of titanium-treated material (H) heated at temperatures below 800° F showed only superficial stains after 1200 hours in the spray (fig. 12). The specimen heated at 800° F was considerably stained, but

showed only slight pitting at the rounded end. Specimens heated at 900° and 1100° F showed some etched and pitted areas along the sides as well as at the ends. The specimen heated at 1000° F showed no evidence of corrosion. The corrosive attack on specimens heated at 1200° and 1400° F was very slight.

Specimens of columbium-treated (K) material heat-treated at different temperatures showed no marked differences in resistance to corrosion by the salt spray (fig. 13). All of them exhibited only superficial stains traceable to slight pitting attacks at the ends of the specimens. On removal of the stain, no evidence of attack was found in other areas.

At the end of 1000 hours' exposure, heat-treated specimens of molybdenum-treated material (M) showed no appreciable staining and no evidence of progressive corrosive attack (fig. 14). The specimen exposed as received and that heated at 1000° F showed slight staining at the end of 24 and 48 hours, respectively. However, this initial staining seemed to be arrested at this point, and no evidence of increased corrosive attack was noted during the test.

It is probable that all heat-treated specimens of the four materials would comply acceptably with any of the usual salt-spray-test requirements except those of E material heated at 1000° and 1100° F and those of H material heated at 900° and 1100° F. On the basis of resistance to corrosion by the salt spray the four materials, as received or heated at temperatures below about 900° F, would be rated in the order E-H-M-K. After heating to higher temperatures (above 900° F), the rating would be in the order K-M-H-E.

Specimens of tie rods of 18:2, 16:1, and K-monel (O, U, and R) after 300 hours' exposure, in the "as received" condition, to the salt-spray test are shown in figure 15. The corrosive attack on these materials in the salt spray was much more severe than with the plain or stabilized 18:8 materials previously discussed. After 300 hours, there were corrosion pits on all three specimens, the most severe being on the 16:1 material. The three materials would probably stand in the following order with respect to resistance to corrosion in the salt spray: 18:2 (O), K-monel (U), and 16:1 (R).

(b) Boiling-Nitric-Acid Test

Specimens (approximately 1 in. in length) of the three tie-rod materials E, H, and K were heated at temperatures ranging from 200° to 1500° F for periods of 30 minutes and 1 hour. Similar specimens of M material were heated 30 minutes at temperatures up to 1900° F. Specimens of E material were also heated at temperatures ranging from 700° to 1000° F for periods of 5, 10, and 24 hours. The specimens were ground on emery paper to remove any scale formed during heat treatment. They were then cleaned and weighed prior to immersion for 3 consecutive 48-hour periods in boiling 65-percent nitric acid under a reflux condenser. The specimens were weighed at the end of each 48-hour period. An example of the weight loss of 18:8 tie-rod specimens, materiel E (heating period 30 minutes) for each 48-hour period, is given in table 8. These results and those obtained on other materials are plotted in figures 6 to 10 and 16 to 18 as the corrosion loss - defined as the loss in original weight, in percent, in 144 hours.

For the 30-minute heating period, the corrosion loss of the straight 18:8 tie-rod material (E) in boiling nitric acid remained constant up to 900° F (fig. 16). Above this temperature, the corrosion loss increased abruptly between 900° and 1100° F, decreased rapidly between 1100° and 1300° F, and again increased between 1300° and 1500° F. Similar abrupt variations in corrosion loss were noted with specimens of titanium-treated material (H). These variations were less marked, however, and occurred at slightly lower temperatures (fig. 17). With the columbium-treated material (K), the changes in corrosion loss at higher temperatures were comparatively small and showed no abrupt variations (fig. 18). The chief effect of increasing the time of heating, as shown by the nitric acid test results, was to cause decreased corrosion resistance at progressively lower temperatures. With the molybdenum-treated material, the only abrupt variation in corrosion loss, occurred at temperatures between 1100° and 1300° F (fig. 10).

The nitric-acid-test results agreed in general with those of the salt-spray test. Both tests indicated a high degree of stability for the columbium-treated material at the higher temperatures, and for the molybdenum-treated material at all temperaturesranges except between 1100° and 1300° F. The other two materials showed comparatively wide variations in corrosion resistance after heating in the temperature range 900° to 1500° F.

Similar variations in corrosion loss in boiling nitric acid were noted in annealed specimens of E material (18:8) (quenched from 2100° F prior to reheating). The results of this test are shown in figure 16 for comparison with those obtained on the cold-worked material. It will be noted that the variations were less pronounced and occurred at higher temperatures.

The losses in weight in boiling nitric acid of 18:2 and 16:1 tie-rod specimens as received are given in table 9.

(c) Boiling-Copper-Sulphate Sulphuric-Acid Test

Specimens of tie-rod materials E, H, and K (size 1/2-20) and material M (size 5/8-18) were heated 30 minutes prior to testing 100 hours in a boiling-copper-sulphate sulphuric-acid solution containing 13 g $CuSo_4 \cdot 5H_2O$ and 47 ml conc. H_2SO_4 per liter. The loss in weight and the change in electrical resistance of the specimens were measured. These data for the E material are recorded in table 10. A significant change in electrical resistance was shown only by the specimen heated at 1100° F. This specimen also suffered a very high weight loss. The specimens heated at 1000° and 1200° F also showed substantial weight losses but no significant changes in resistance. Microscopic examination later showed that all three specimens had been subject to a considerable amount of intergranular penetration. Figure 19 shows an example of the type of attack found on the specimen heated at 1100° F.

With the titanium-treated (H), the columbium-treated (K), and the molybdenum-treated (M) materials, the results of the $CuSO_4$-H_2SO_4 test were entirely negative. The specimens showed no significant changes in electrical resistance and only very small weight losses, which were practically constant, for all temperatures up to 1900° F.

Specimens of 18:2 and 16:1 materials were severely attacked after 100 hours in the boiling-copper-sulphate sulphuric-acid solution. The weight losses for both materials varied from 30 to 100 percent. These results furnished no information except that the materials were soluble in the acid solution used.

THE EFFECT OF HEAT TREATMENT ON THE MICROSTRUCTURE OF THE MATERIALS

The microstructure representative of the SAE 1050 steel tie-rod materials is shown in figure 20 A. On heating for a 30-minute period specimens of this material showed no appreciable change in structure below 1000° F, at which temperature the first definite evidence of recrystallization appeared (fig. 20 B).

The microstructures of E material (18-8) heated 30 minutes in the temperature range 900° to 1500° F are shown in figures 21 and 22. The specimens were given a light electrolytic etch in 10-percent oxalic acid to show carbide precipitation. No significant changes in structure were noted in specimens heated at temperatures up to and including 900° F. At 1000° F, however, the material showed a considerable amount of carbide precipitation on the grain boundaries and slip planes (fig. 21 B). At temperatures above 1200° F precipitation of carbides on the slip planes appeared to diminish. This was probably due to the beginning of recrystallization. In the specimen heated at 1300° F recrystallization was well advanced.

The variations in corrosion resistance previously noted in specimens of E material heated at temperatures in the range 900° to 1500° F may be due, in part at least, to the structural changes which occur at these temperatures. Thus, the sudden decrease in corrosion resistance at about 1000° F can be associated with the precipitation of chromium carbide which caused the material surrounding the carbide particles to become deficient in chromium. At temperatures below the recrystallization range the precipitation was confined to the slip planes and grain boundaries of relatively large grains elongated by cold work. The corrosive attack in specimens heated in this temperature range was severe, particularly at the ends where the grain boundaries and slip planes were most exposed. Examples of the type of corrosive attack in boiling nitric acid found at the ends of heat-treated specimens of materials E, H, K, and M are shown in figure 23. Figure 23 (E shows an example of corrosive attack on the side of a heat-treated specimen. The first effect of recrystallization apparently was to cause a temporary increase in corrosion resistance by the formation of a large number of very small grains, which thus broke up the

paths by which the corrosive attack could penetrate into the metal. This probably is associated with the temporary improvement noted in the corrosion resistance of specimens heated 30 minutes at 1200° and 1400° F in the salt-spray test (fig. 11) and in the boiling-nitric-acid test (fig. 16). At higher temperatures (up to 1500° F) this effect was probably diminished by grain growth. At still higher temperatures, the corrosion resistance undoubtedly would again increase as a result of the solution of the carbide particles and the diffusion of chromium.

The variations observed in the curve (fig. 16) showing the corrosion loss in nitric acid of annealed specimens of E material may be explained in a somewhat similar manner. Microscopic examination of these specimens showed (fig. 24) that the carbide precipitation, which began at 1100° F, was at first entirely confined to the grain boundaries. At 1400° F, however, precipitation began to appear also on the twinning planes (fig. 24 E). This, in effect, was equivalent to a sudden substantial decrease in grain size and resulted in a temporary improvement in corrosion resistance.

The effect of titanium, columbium, or molybdenum additions to straight 18:8 stainless steel in increasing its corrosion resistance at high temperatures usually is attributed, in large measure, to the affinity of these elements for carbon. (See references 9, 10, 11, 12, and 13.) Heating of material containing these elements in sufficient quantities causes precipitation of titanium, columbium, or molybdenum carbides in preference to chromium carbide. The material surrounding carbide particles is therefore not impoverished in chromium and its corrosion resistance is therefore not lowered. Moreover, with these added elements, the carbides are, in general, more widely distributed. It is probable that the grain refinement resulting from the addition of titanium, columbium, or molybdenum also increases the corrosion resistance of the stabilized materials.

The titanium-treated (H) material, as shown in figures 25 and 26, contained a considerable amount of delta ferrite which, except for precipitation, remained unchanged throughout the heat treatment (up to 1500° F). The material in the cold-worked state was quite magnetic and apparently contained an appreciable amount of ferrite produced by cold-working. Precipitation, probably of titanium carbide, began at about 800° F. Between 1100°

and 1200° F another type of precipitation appeared. The second precipitate apparently occurred in segregated areas, producing a banded structure. The banding is believed to be due largely to chromium-carbide precipitation in segregated areas containing relatively high carbon or little titanium. The segregated bands of heavy precipitation appeared to be areas of lower corrosion resistance (fig. 23 B).

The columbium-treated (K) material also displayed a considerable amount of segregation (figs. 27 and 28). In this material, however, the precipitation in the segregated areas was not so heavy. At lower temperatures (800° to 1100° F) the material contained a widely distributed precipitate which is believed to be columbium carbide. No delta ferrite was noted in the material. However, in the cold-worked state, the material was somewhat magnetic and apparently contained some ferrite produced by cold-working.

No significant changes in structure were noted in specimens of molybdenum-treated material (M) heated at temperatures up to and including 1100° F (figs. 29 and 30). Heavy carbide precipitation appeared at 1200° F (fig. 29 D). At higher temperatures the carbides became somewhat larger, but decreased gradually in number and disappeared entirely at 1800° F, at which temperature recrystallization was practically complete. The molybdenum-treated material contained stringers or chains of delta ferrite which, except for carbide precipitation, remained unchanged throughout the heat treatment. In boiling nitric acid, corrosive attack at the ends of specimens of this material was particularly severe along these stringers or chains (fig. 23 D).

The microstructures of 18:2, 16:1, and K-monel materials as received are shown in figures 31 to 33. The cold-worked structure produced by forming operations had not been completely eliminated by the heat treatment applied to these materials after fabrication. Much free chromium ferrite was found to be present as stringers or chains in materials 18:2 and 16:1.

Transverse sections of severely cold-worked tie-rod materials were examined for evidence of the crossed bands often noted during metallographic examinations of such materials. A typical X-band structure in an 18:8 tie rod is shown in figure 34. Similar X bands were found in all the severely cold-worked materials examined (materials A,

B, C. D, E. F, G, H, J, K, and M). Well defined X bands were also found in K-monel tie rods (materials U and V), but none were detected in 18:2 or 16:1 tie rods (materials O, P, R, and S).

It is generally believed that these bands are zones in which the metal has been more severely cold-worked during fabrication than in zones outside the bands. Evidence based on the microstructure of some of the materials and on Vickers indentation tests supports this view.

The typical microstructure on transverse sections within and outside the X bands in a specimen of an 18:8 stainless steel tie rod are shown in figures 35 and 36. Comparison of the size of grains in these micrographs shows that they are smaller and more uniformly deformed within the X band than in areas outside. Vickers indentation tests made on a transverse section of a tie rod of M material (18:8 Mo) showed that the metal within the bands was distinctly harder than that outside. The average value of readings obtained within the bands was 423 (Vn-30). Outside the bands the average value was 388.

Although K-monel tie rods also showed X bands, the Vickers indentation number of metal within the bands was not appreciably higher than that outside. It is probable that the heat treatment received by this material after fabrication caused the hardness to become more uniform, even though the material was not completely recrystallized to remove all evidence of cold work. In this connection it was noted that complete recrystallization during annealing of any of the materials caused disappearance of the X bands.

DISCUSSION OF RESULTS

The test results indicated that a large part of the residual stress in SAE 1050 steel tie rods could be relieved by low-temperature heat treatment without difficulty. Heating for 30 minutes at 600° F, for instance, relieved about two-thirds of the stress in the 3/8-24 size tie rod without lowering the important mechanical properties of the material. Since this type of tie rod depends for protection upon a cadmium coating applied after fabrication, there should be no objection to heat treatment in regard to effect on corrosion resistance.

The tensile-test results indicated that the stainless steel tie rods of the straight 18:8 composition or the same stabilized by titanium, columbium, or molybdenum additions could be heated 30 minutes at temperatures up to 1000° F without seriously lowering the mechanical properties. At this temperature most of the residual stress would be relieved. The corrosion resistance in laboratory corrosion tests of some of these materials, however, appeared to be impaired after heating at temperatures above 800° or 900° F.

The interpretation of laboratory corrosion-test results is difficult because the conditions of testing usually have little relation to actual service conditions.

Of the various tests used, the salt-spray test probably most nearly approaches service conditions, particularly if the materials are to be used in a marine environment. In this test, however, the corrosive attack is too slow and too slight to permit quantitative measurements of corrosion to be made. The interpretation of results is therefore a matter of opinion. With the boiling-nitric-acid test accurate, reproducible, quantitative measurements may be obtained, but the conditions of testing have no relation whatever to service conditions. The test is of some value, however, in comparing the effect of the changes in structure produced by heat treatment. The boiling-copper-sulphate sulphuric-acid test is generally recommended to show up susceptibility to intercrystalline corrosion; but with the samples used in the present investigation, the test was found to be not very sensitive.

The results of the laboratory corrosion tests indicated that tie rods of 18:8 corrosion-resistant steel should not be heated at temperatures above 900° F since material heated above this temperature failed in all three corrosion tests. A 30-minute heat treatment at 900° F could be expected to relieve at least 40 percent of the residual stress. This might be of considerable importance where tie rods of very high residual stress are concerned. An actual service test has been made on two 18:8 corrosion-resistant tie rods heated 30 minutes at 900° F. After 800 hours flying time in a flying boat, the tie rods showed no evidence of corrosion and no signs of fatigue cracks or other damage.

Judging from the results obtained on the material supplied for the present investigation, one would conclude that the heat treatment of titanium-treated tie rods at

temperatures above 800° F could not be recommended. Specimens of this material heated at higher temperatures failed in the salt-spray and the boiling-nitric-acid test. The failure of this material may have been due partly to segregation, and it is possible that a material of more uniform structure might prove satisfactory after heat treatment at higher temperatures.

The columbium-treated material satisfactorily withstood all three of the laboratory corrosion tests after heat treatment at temperatures up to and including 1000° F although this material also exhibited segregation.

The molybdenum-treated material also exhibited improved stability over a wide range of temperature. The corrosion resistance of this material was appreciably impaired only when heated at temperatures between 1100° and 1300° F. The tests indicate that either the columbium- or the molybdenum-treated materials could be heated 30 minutes with safety at any temperature up to 1000° F. At this temperature most of the residual stress would be relieved.

It has been pointed out previously that the most severe corrosive attacks, both in the salt spray and in the boiling nitric acid, were localized at the exposed ends of specimens, in segregated areas of heavy carbide precipitation, along exposed grain boundaries and slip planes, or along stringers of inclusions. In service most of these areas would not be exposed. It is believed, therefore, that any conclusions drawn from the corrosion-test results will be in error on the side of safety.

CONCLUSIONS

Heat treatment for relief of residual stress in streamline tie rods of various compositions was investigated with regard to:

1. The effect of heat treatment on the relief of residual stress

2. The effect of heat treatment on the physical properties of the materials

3. The effect of heat treatment on the corrosion resistance of the materials

4. The effect of heat treatment on the microstructure of the materials.

The results of the investigation indicated that about two-thirds of the residual stress in tie rods of SAE 1050 steel could be relieved by heating 30 minutes at 600° F. This treatment did not materially lower the mechanical properties of the material.

Tie rods of stainless steel of straight 18:8 composition or of the same with additions of titanium, columbium, or molybdenum could be heated at temperatures up to 1000° F without seriously lowering the mechanical properties of the materials. At this temperature, most of the residual stress would be relieved.

Specimens of 18:8 and titanium-treated 18:8 materials exhibited impaired corrosion resistance in laboratory corrosion tests when heated 30 minutes at temperatures above 900° and 800° F, respectively.

Tie rods of columbium-treated and molybdenum-treated 18:8 steel displayed stability over wider ranges of temperature. Both materials could be heated 30 minutes at temperatures up to and including 1000° F without serious detriment to the corrosion resistance. At this temperature, most of the residual stress would be relieved.

Heat-treated tie rods of 18:2, 16:1, and K-monel materials were found to contain very low residual stress in the "as received" condtion. Further heat treatment for relief of stress would not be necessary. These materials were somewhat inferior to the austenitic stainless steels in corrosion resistance in the laboratory corrosion tests used in this investigation.

The authors wish to acknowledge their indebtedenss to Dr. D. J. McAdam, Jr., who developed the formulas used in estimating residual stress, to C. S. Aitchison and R. W. Mebs, who made the tensile tests, and to H. L. Logan, who performed some of the work on X bands.

National Bureau of Standards,
Washington, D. C., August 14, 1941.

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TABLE 2.- PARTIAL RESIDUAL STRESS NEAR CENTER OF MAJOR AXIS OF SAE 1050 STEEL (MATERIALS A AND B) TIE RODS AS RECEIVED

[Measurements made on specimens split by a saw cut]

Material		Original width (in.)	Width after splitting (in.)	Change in width, 2a (in.)	Length of cut, l (in.)	Width of segment (in.)	Radius of curvature (in.)	Partial residual stress (tension), S_1 (lb/sq in.)
SAE 1050 steel	A1	0.551	0.532	0.019	1.28	0.26	86.2	38,300
	A2	.550	.523	.027	1.38	.26	70.5	46,800
	A3	.550	.523	.027	1.37	.26	69.5	47,500
	[a]A4	.551	.547	.004	.50	.26	62.5	48,600 average
			.545	.006	.64	.26	68.3	
			.540	.011	.87	.26	68.8	
			.535	.016	1.05	.26	68.9	
			.527	.024	1.28	.26	68.3	
			.517	.034	1.55	.26	70.6	
SAE 1050 steel	B1	.547	.524	.023	1.48	.25	95.2	33,100
	B2	.547	.525	.022	1.45	.25	95.5	32,900
	B3	.546	.524	.022	1.43	.25	92.9	33,900
	[a]B4	.547	.544	.003	.51	.25	86.7	34,700 average
			.542	.005	.68	.25	92.5	
			.536	.011	1.00	.25	91.0	
			.530	.017	1.24	.25	90.5	
			.525	.022	1.42	.25	91.7	
			.517	.030	1.65	.25	90.7	

[a]Measurements were made at various lengths of cut on specimens A4 and B4 to test the accuracy of method of calculation.

TABLE 3.- PARTIAL RESIDUAL STRESS AT END OF MINOR AXIS OF TIE RODS AS RECEIVED, OF MATERIALS A AND B

[Measurements made on specimens machined to half of original thickness]

Material		Original thickness (in.)	Thickness after machining (in.)	Deflection in 4-inch gage length (in.)	Radius of curvature (in.)	Partial residual stress (tension), S_2 (lb/sq in.)
SAE 1050 steel	A	0.138	0.071	0.086	23.2	53,300
	B	.139	.070	.088	22.7	53,600

TABLE 5.- EFFECT OF TEMPERATURE AND PERIOD OF HEATING ON RELIEF OF RESIDUAL STRESS IN SIZE 3/8-24 SAE 1050 STEEL TIE-ROD SPECIMENS

[Material A]

Heating temperature (°F)	Residual stress after various heating periods (tension) (lb/sq in.)			
	30 minutes		2 hours	
	Partial, S_1	Residual, S	Partial, S_1	Residual, S
As received	45,300	98,700	[a]------	[a]------
200	43,500	94,900	[a]------	[a]------
300	34,200	74,500	[a]------	[a]------
400	27,000	58,800	28,400	61,900
500	21,900	47,700	[a]------	[a]------
600	17,800	38,800	6,200	13,500
700	6,600	14,400	0	0
800	3,000	6,500	0	0
900	0	0	0	0

[a]No measurements were made.

TABLE 4.- RESIDUAL STRESS AT END OF MINOR AXIS OF TIE RODS AS RECEIVED

[+ sign indicates tension; - sign, compression; * , no appreciable deflection]

Material		Partial residual stress, S_1	Partial residual stress, S_2	Residual stress, $S = S_1 + S_2$	Ratio, S_1/S
SAE 1050 steel	A	+45,300	+53,300	+98,600	0.46
	B	+33,600	+53,600	+87,200	.39
18:8 steel	C	+49,100	+79,100	+128,200	.38
	D	+55,100	+65,000	+120,100	.46
	E	+78,000	+75,000	+153,000	.51
	F	+47,400	+32,600	+80,000	.59
18:8 Ti	G	+36,400	+33,200	+69,600	.52
	H	+45,200	+58,600	+103,800	.44
	I	+40,600	+71,200	+111,800	.36
18:8 Cb	J	+42,200	+42,300	+84,500	.50
	K	+49,900	+71,300	+121,200	.41
	L	+33,900	+71,000	+104,900	.32
18:8 Mo	M	+55,000	+57,500	+112,500	.49
18:2 steel	N	-19,400	*0	-19,400	
	O	-4,500	*0	-4,500	
	P	-4,900	*0	-4,900	
16:1 steel	Q	-19,400	*0	-19,400	
	R	-8,700	*0	-8,700	
	S	-7,400	*0	-7,400	
K-monel	T	-3,600	*0	-3,600	
	U	+7,400	+10,100	+17,500	.42
	V	+10,800	+12,900	+23,700	.46

TABLE 6.- EFFECT OF TEMPERATURE AND PERIOD OF HEATING ON RELIEF OF RESIDUAL STRESS IN SIZE 1/2-20 18:8 STAINLESS-STEEL TIE-ROD SPECIMENS

[Material E]

Heating temperature (°F)	Residual stress after various heating periods (tension) (lb/sq in.)											
	30 minutes		1 hour		2 hours		5 hours		10 hours		24 hours	
	Partial, S_1	Residual, S	Partial, S_1	Residual, S	Partial, S_1	Residual, S	Partial, S_1	Residual, S	Partial, S_1	Residual, S	Partial, S_1	Residual, S
As received	78,000	153,000	78,000	153,000	78,000	153,000	78,000	153,000	78,000	153,000	78,000	153,000
300	78,900	155,000	69,200	136,000	64,200	126,000	[a]-----	[a]------	[a]-----	[a]------	[a]-----	[a]------
500	68;600	135,000	66,300	130,000	64,200	126,000	[a]-----	[a]------	[a]-----	[a]------	[a]-----	[a]------
700	65,100	128,000	62,300	122,000	64,300	126,000	59,300	116,000	57,300	112,000	57,200	112,000
800	58,200	114,000	54,200	106,000	55,100	108,000	49,000	96,100	50,400	99,000	46,600	91,000
850	52,100	102,000	47,400	93,000	45,500	89,000	37,300	73,200	31,100	61,000	30,600	60,000
900	45,900	90,000	39,800	78,000	36,100	71,000	26,900	52,800	17,800	35,000	10,800	21,000
950	40,500	79,000	33,100	65,000	24,100	47,000	[a]-----	[a]------	[a]-----	[a]------	[a]-----	[a]------
1000	31,100	61,000	24,100	47,000	15,300	30,000	[a]-----	[a]------	[a]-----	[a]------	[a]-----	[a]------
1050	17,700	35,000	17,800	35,000	8,900	17,000	[a]-----	[a]------	[a]-----	[a]------	[a]-----	[a]------
1100	12,300	24,000	9,200	18,000	5,500	11,000	[a]-----	[a]------	[a]-----	[a]------	[a]-----	[a]------
1200	4,700	9,000	0	0	0	0	[a]-----	[a]------	[a]-----	[a]------	[a]-----	[a]------
1400	0	0	0	0	0	0	[a]-----	[a]------	[a]-----	[a]------	[a]-----	[a]------

[a]No measurements were made.

TABLE 7.- TENSILE PROPERTIES OF SIZE 5/8-18 TIE-ROD SPECIMENS OF MATERIALS M, P, S, AND V, AS RECEIVED

Material	Ultimate tensile strength (lb/sq in.)	Yield strength (offset = 0.2 percent) (lb/sq in.)	Permanent set in (0.0002 in./in.) (lb/sq in.)	Elongation (2 in.) (percent)	Location of fracture
M (18:8 Mo)	196,100	139,000	145,000	-------	Outside elongation marks
	189,100	140,000	145,000	7.0	Free length
P (18:2)	216,100	168,500	169,000	15.0	Free length
	211,600	168,000	166,000	17.0	Free length
S (16:1)	195,000	152,500	169,000	14.5	Free length
	192,900	153,000	159,000	13.0	Free length
V (K-monel)	189,100	175,000	158,000	10.0	Free length
	189,500	175,000	177,000	11.0	Free length

TABLE 9.- CORROSION RESISTANCE IN BOILING NITRIC ACID (65 PERCENT) OF 18:2 AND 16:1 TIE-ROD SPECIMENS AS RECEIVED

Material		Heating temperature ($^{\circ}$F)	Original weight (grams)	Weight loss in boiling 65-percent nitric acid				
				First 48 hours (gram)	Second 48 hours (gram)	Third 48 hours (gram)	Total loss (144 hr.) (gram)	Total loss (144 hr.) (percent)
18:2	N	As received	1.5547	0.0438	0.5020	0.5070	0.1447	9.31
	O	----do------	14.2812	.1798	.2377	.2746	.6921	4.85
	P	----do------	24.1737	.1884	.2929	.3867	.8680	3.59
16:1	Q	----do------	1.6965	.0757	.0824	.0815	.2396	14.59
	R	----do------	14.8570	.2131	.2351	.2328	.6810	4.58
	S	----do------	19.0676	.2292	.2628	.2631	.7551	3.96

TABLE 8.- CORROSION RESISTANCE OF HEAT-TREATED TIE-ROD SPECIMENS IN BOILING 65-PERCENT NITRIC ACID. HEATING PERIOD, 30 MINUTES

[Material E (18:8)]

Heating temperature (°F)	Original weight (grams)	Loss in weight in boiling 65-percent nitric acid				
		First 48 hr period (gram)	Second 48 hr period (gram)	Third 48 hr period (gram)	Total loss (144 hr) (gram)	Total loss (144 hr) (percent)
As received	12.8327	0.0111	0.0112	0.0115	0.0338	0.26
200	12.6872	.0105	.0110	.0113	.0328	.26
300	12.4127	.0104	.0105	.0111	.0320	.26
400	12.7235	.0103	.0106	.0111	.0320	.26
500	13.1296	.0115	.0109	.0116	.0340	.26
600	12.7686	.0115	.0128	.0140	.0383	.29
700	12.5296	.0103	.0120	.0132	.0355	.28
800	13.3142	.0111	.0128	.0146	.0385	.29
900	13.2415	.0113	.0138	.0153	.0404	.30
1000	13.0322	.0497	.1229	.1353	.3079	2.36
1100	12.6806	.0819	.1742	.2170	.4731	3.74
1200	12.7521	.0307	.0692	.0906	.1905	1.50
1300	13.0871	.0157	.0331	.0704	.1192	.91
1400	12.3378	.0220	.0743	.0979	.1942	1.56
1500	12.9007	.0524	.1415	.1715	.3654	2.83

TABLE 10.- WEIGHT LOSS AND CHANGE IN ELECTRICAL RESISTANCE OF TIE-ROD SPECIMENS TESTED 100 HOURS IN BOILING-COPPER-SULPHATE SULPHURIC-ACID SOLUTION HEATING PERIOD, 30 MINUTES

[Material E (18:8)]

Heating temperature (°F)	Original weight (grams)	Original resistance (ohm)	Weight loss (grams)	Change in resistance (ohm)
As received	13.6683	0.000134	0.0012	+0.000001
300	14.1208	.000139	.0014	-.000002
400	13.8602	.000134	.0013	-.000004
500	13.4849	.000132	.0014	+.000003
600	13.4794	.000137	.0013	None
700	13.6355	.000140	.0012	+.000002
800	13.3136	.000140	.0012	-.000001
900	13.8518	.000135	.0011	+.000002
1000	13.4651	.000136	.4617	+.000006
1100	13.3988	.000133	3.3878	+.000077
1200	13.4827	.000130	.1234	+.000002
1300	13.3236	.000131	.0025	-.000004
1400	13.5384	.000134	.0026	-.000002
1500	13.7345	.000130	.0011	None
1600	13.4141	.000133	.0010	None
1700	13.1360	.000135	.0010	-.000005
1800	13.3608	.000134	.0013	-.000004
1900	13.5689	.000136	.0021	-.000002

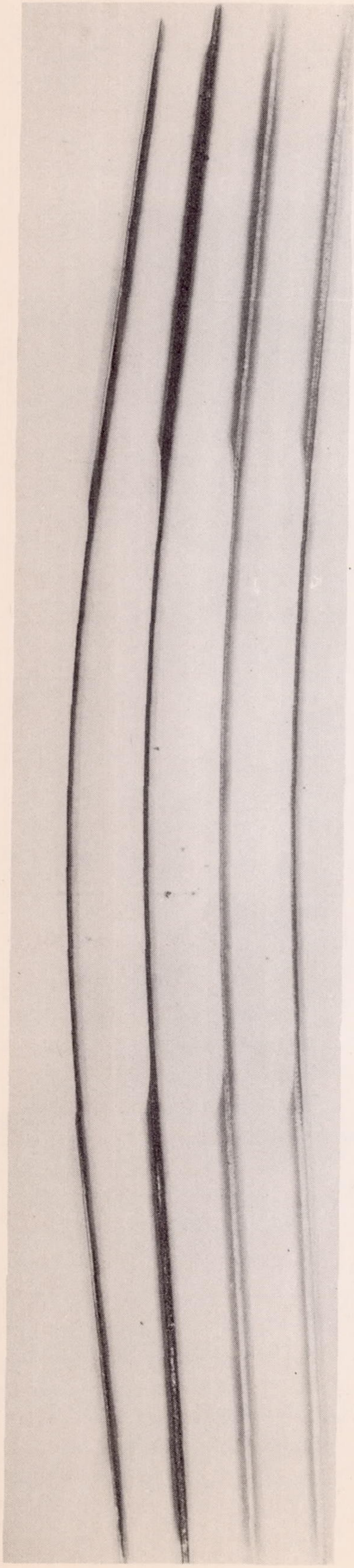
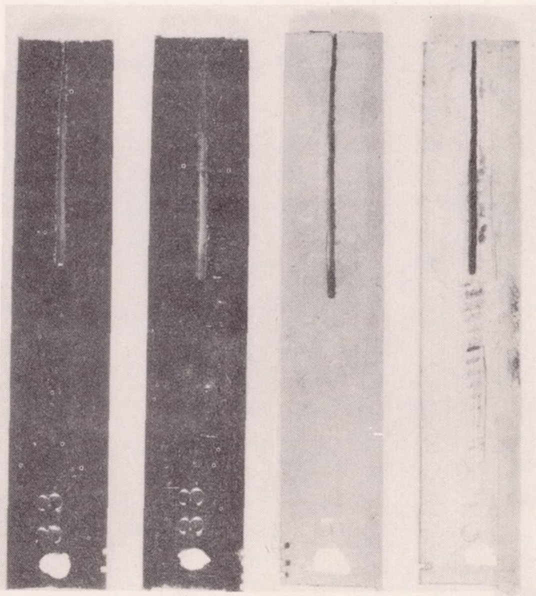


Figure 2.- Distortion in tie-rod specimens caused by partial relief of residual stress. First, second, fifth and sixth specimens from top are 18-8 stainless steel, others are 1050 steel. x 1



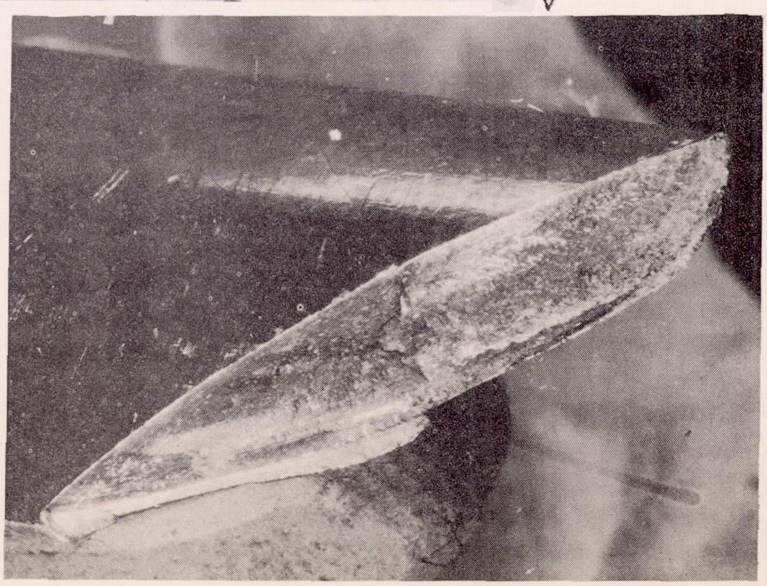


Figure 1.- Torsional fatigue fracture in 18-8 stainless steel tie-rod, x 5.

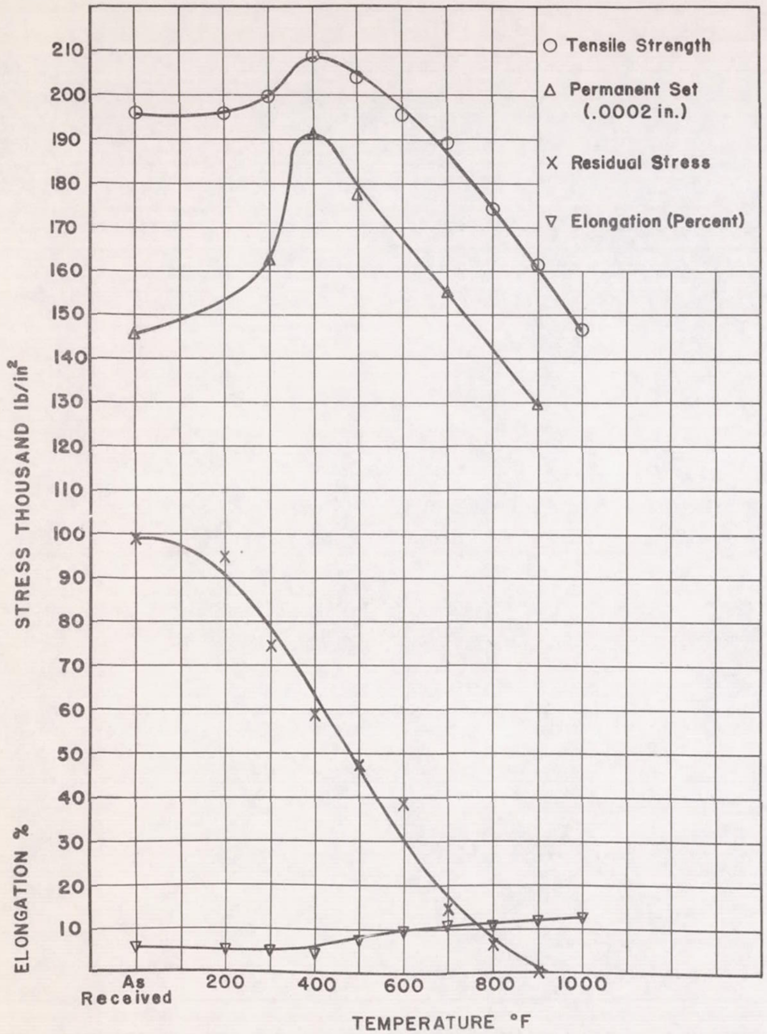


Figure 3.- 1050 steel (Material A) - Effect of heat treatment on physical properties and relief of residual stress. Heating period, 30 minutes.

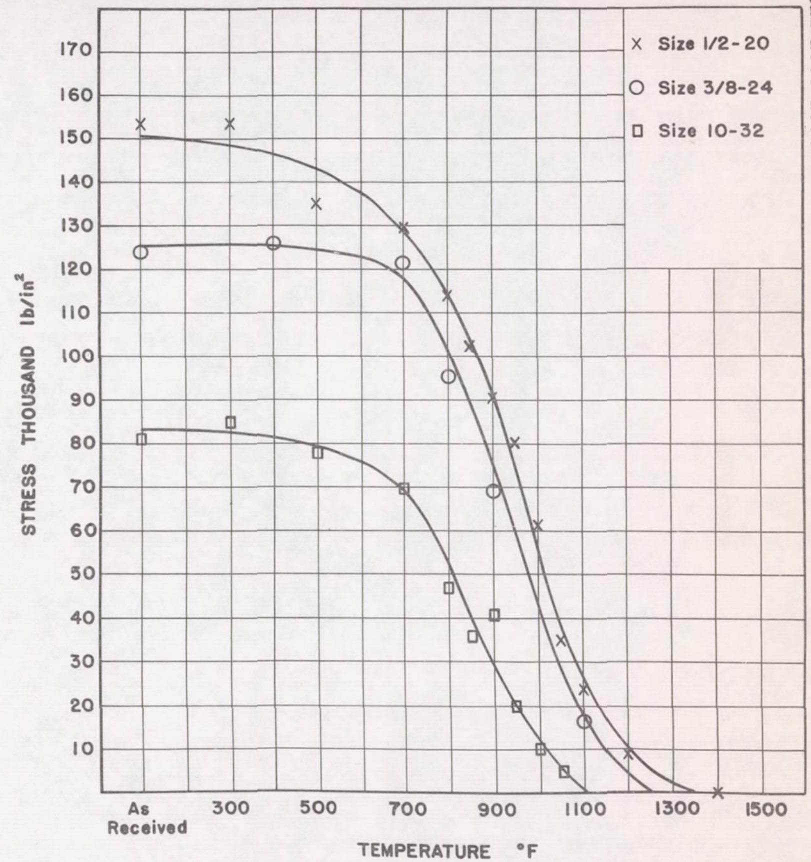


Figure 4.- 18-8 (Materials C,E and F) - Effect of heat treatment on relief of stress in tie-rods of different sizes. Heating period, 30 minutes.

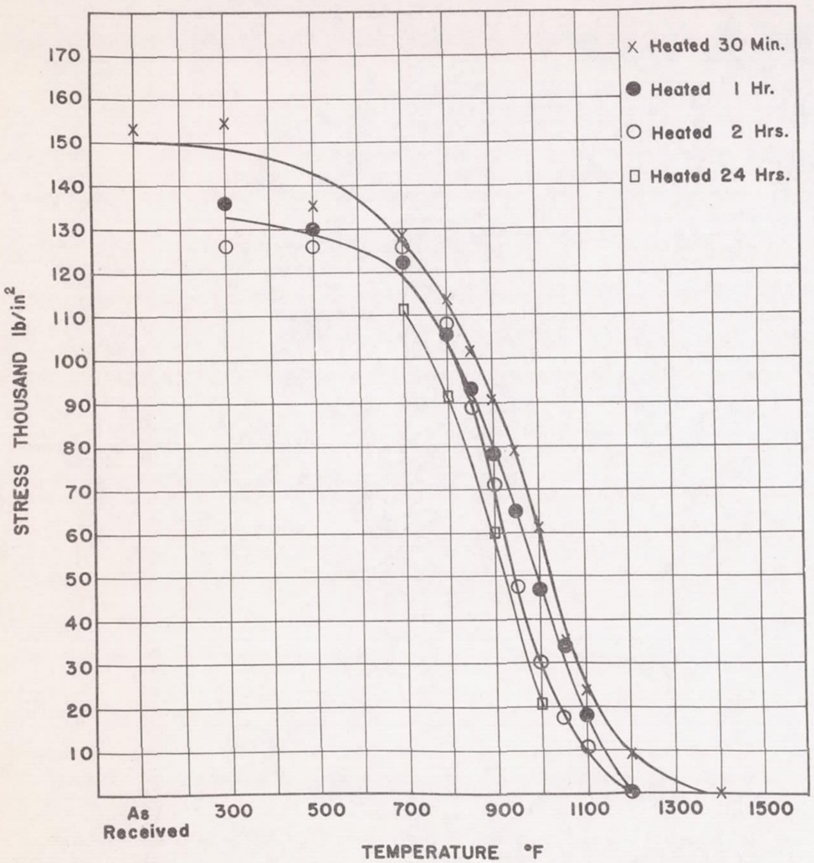


Figure 5.- 18-8 (Material E) - Effect of temperature and period of heating on relief of residual stress.

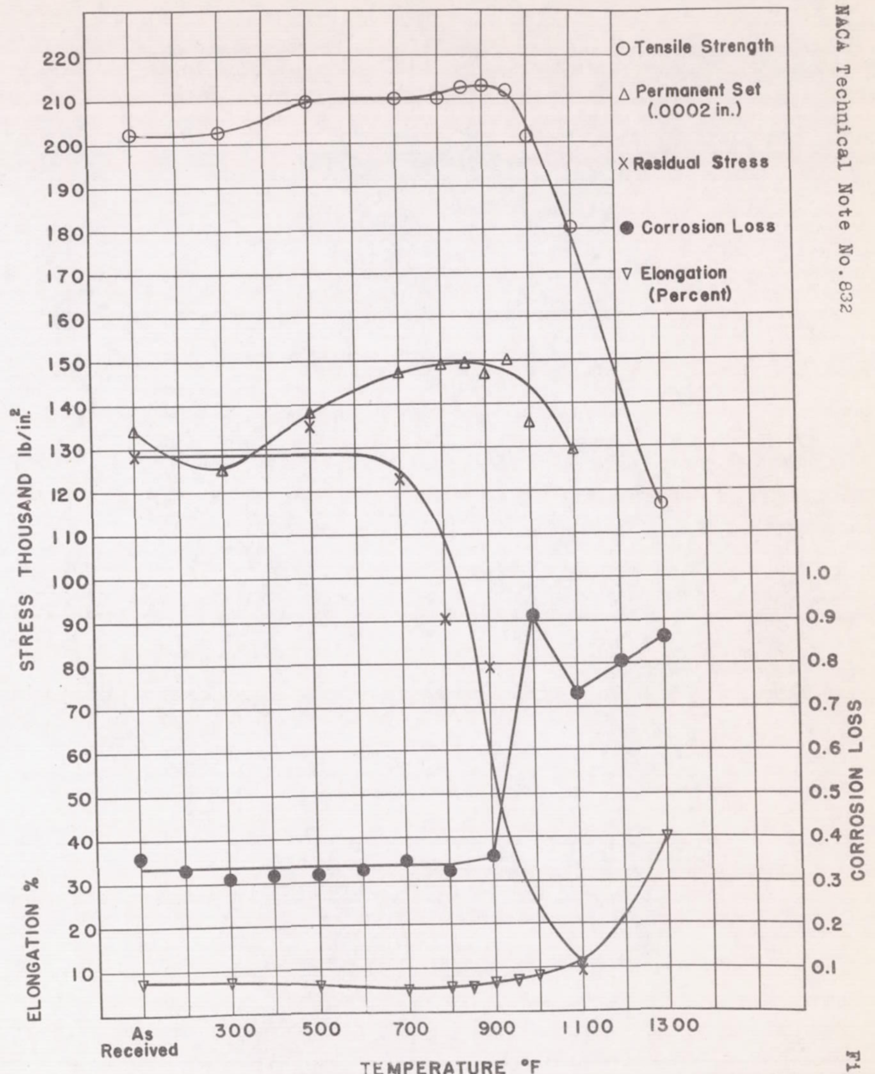


Figure 6.- 18-8 (Material C) - Effect of heat treatment on physical properties, relief of residual stress, and corrosion resistance in boiling nitric acid. Heating period, 30 minutes.

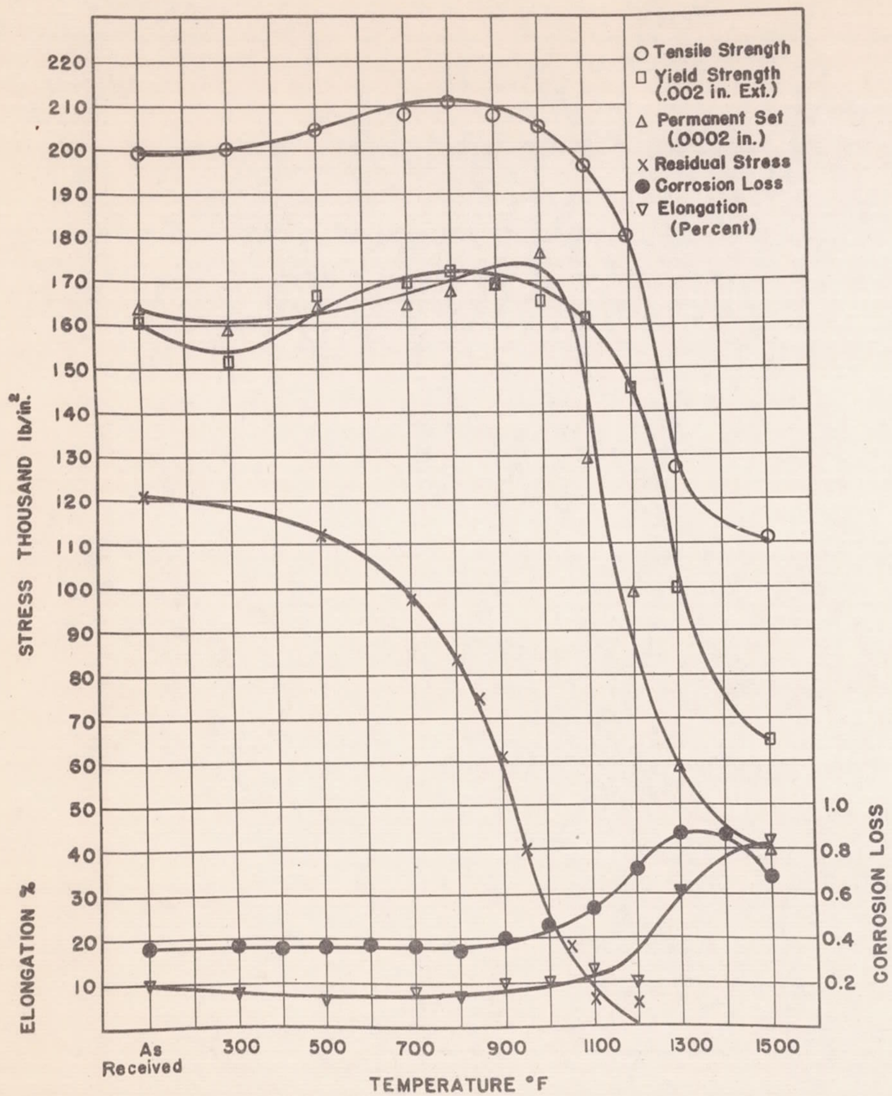


Figure 7.- 18-8-Cb (Material K) - Effect of heat treatment on physical properties, relief of residual stress and corrosion resistance in boiling nitric acid. Heating period, 30 minutes.

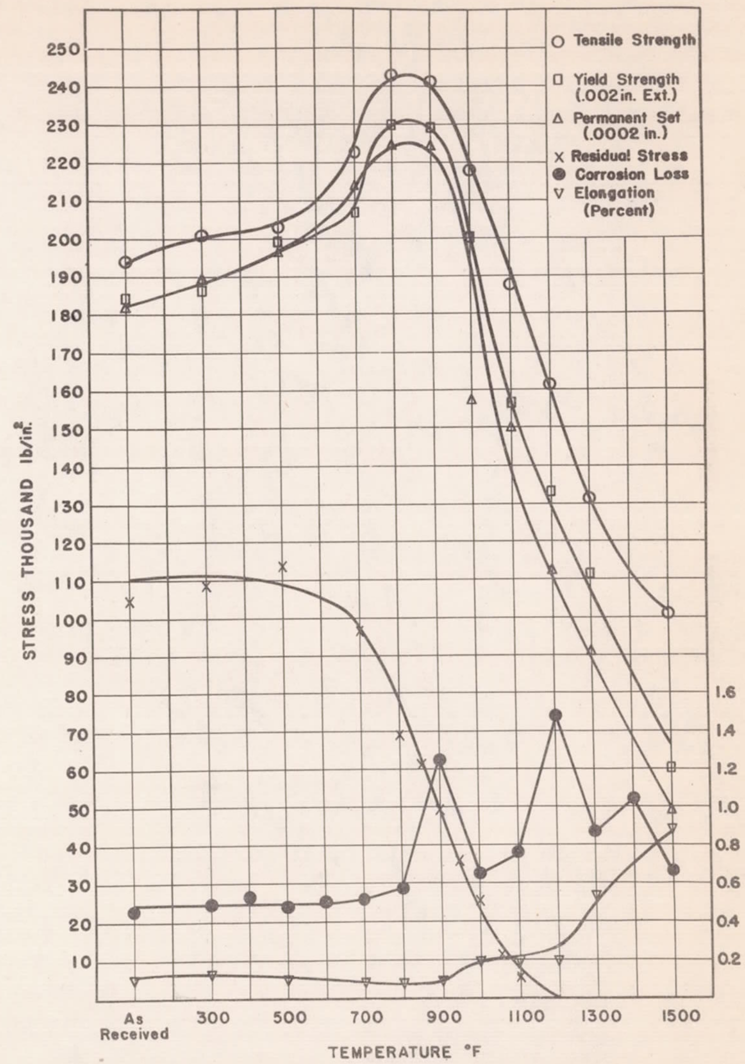


Figure 8.- 18-8-Ti (Material H) - Effect of heat treatment on physical properties, relief of residual stress and corrosion resistance in boiling nitric acid. Heating period, 30 minutes.

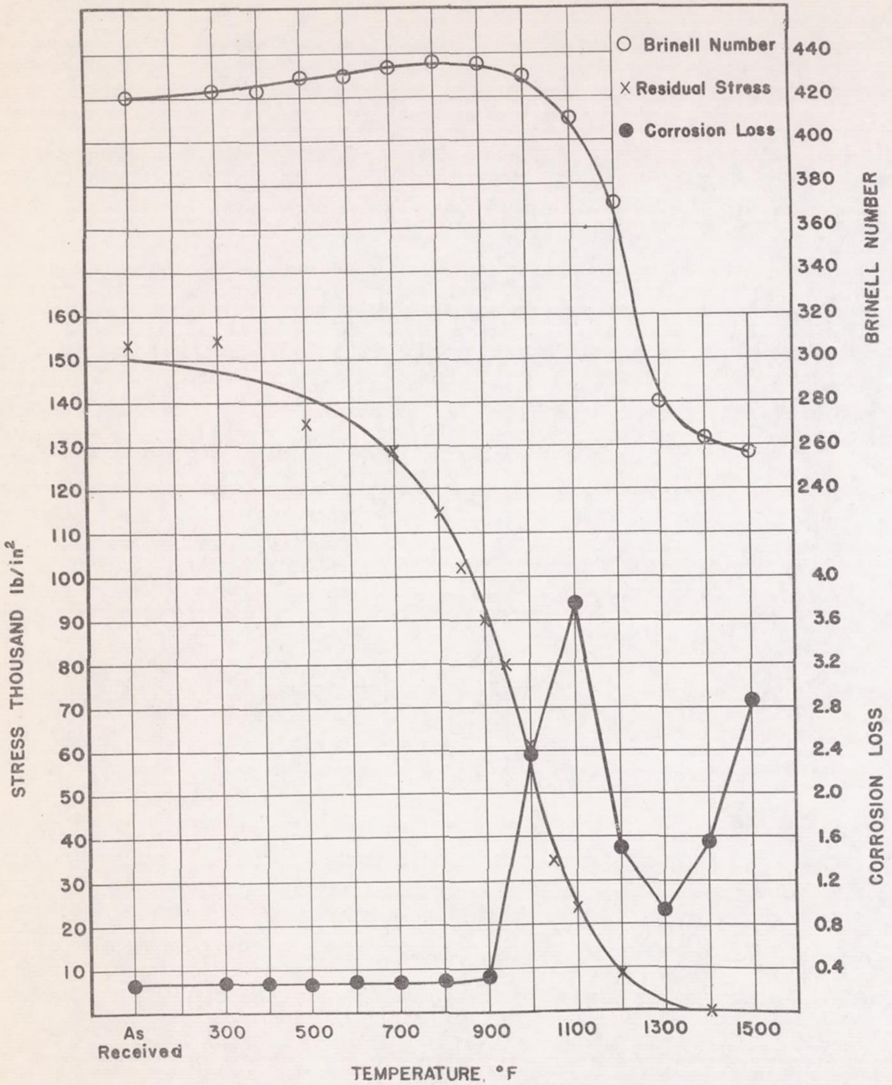


Figure 9.- 18-8 (Material E) - Effect of heat treatment on Brinell number, relief of residual stress and corrosion resistance in boiling nitric acid. Heating period, 30 minutes.

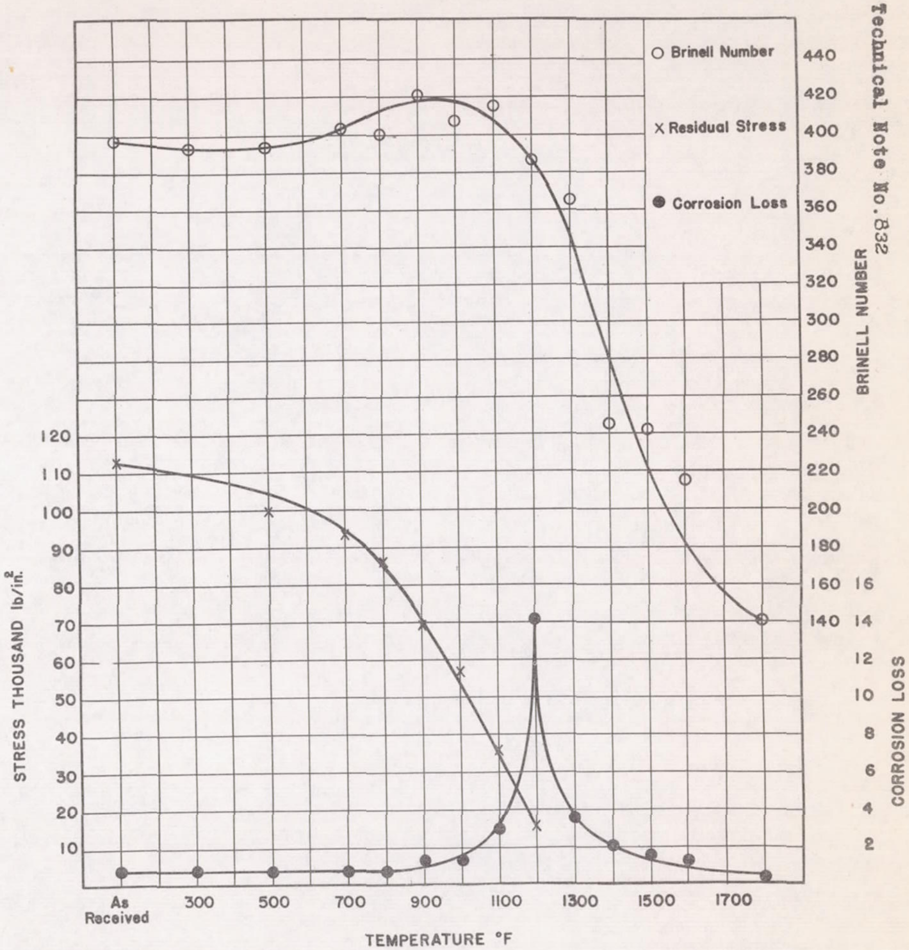


Figure 10.- 18-8-Mo (Material M) - Effect of heat treatment on Brinell number, relief of stress and corrosion resistance in boiling nitric acid. Heating period, 30 minutes.

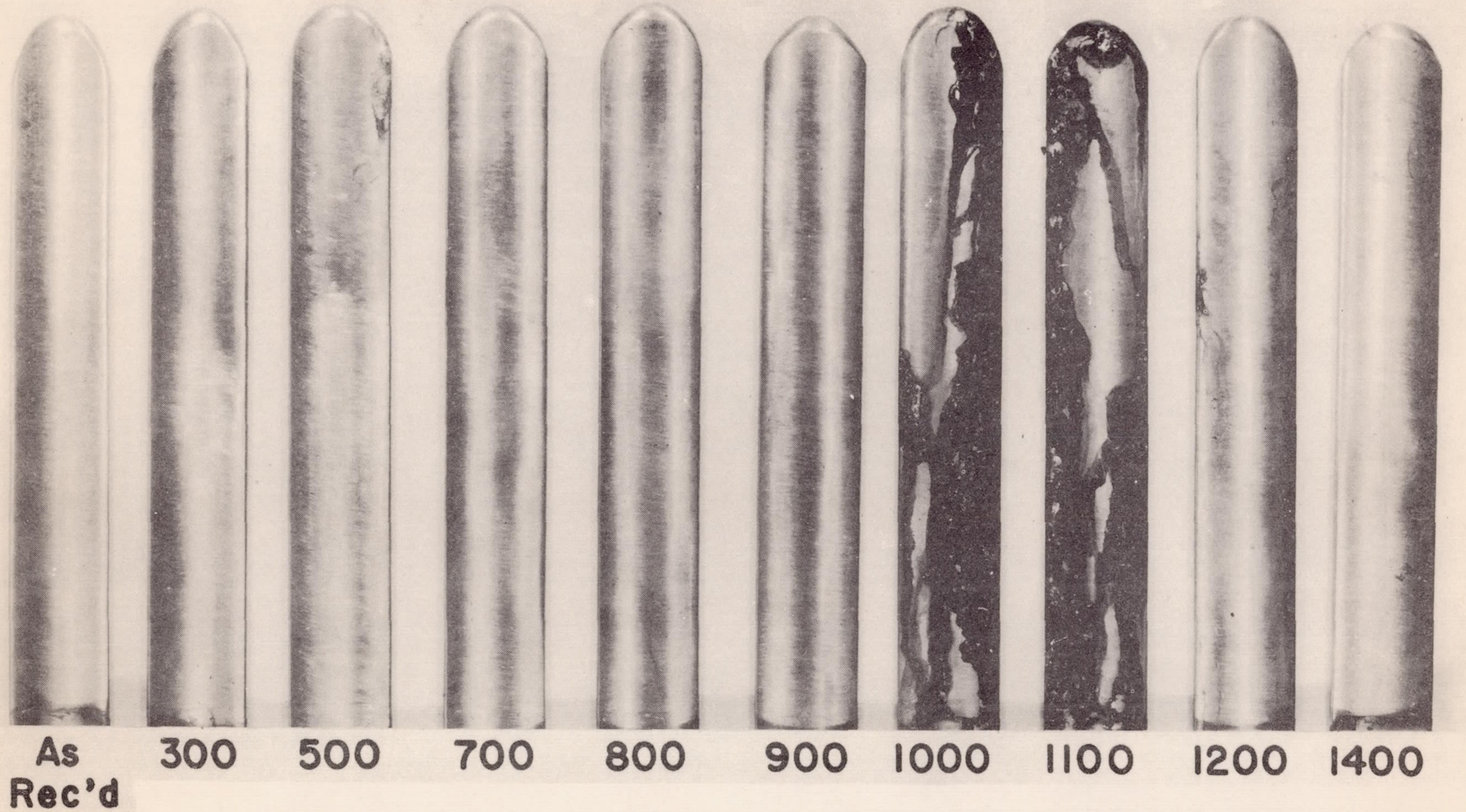


Figure 11.- 18-8 (Material E) - Heat-treated specimens after 1200 hours in salt spray (20 percent). Heating period, 30 minutes. x 7/8

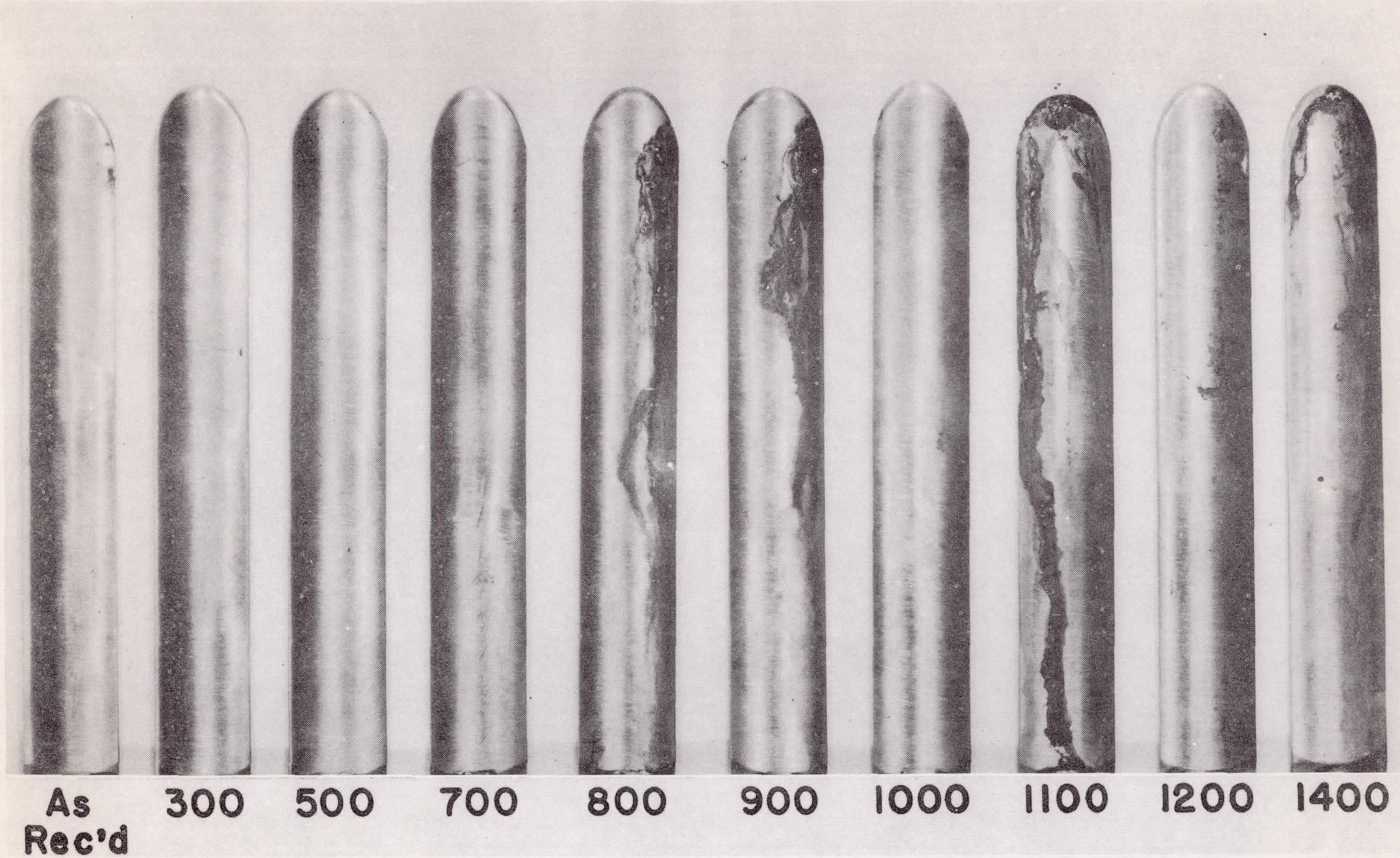


Figure 12.- 18-8-Ti (Material H) - Heat-treated specimens after 1200 hours in salt spray (20 percent).Heating period, 30 minutes. x 7/8

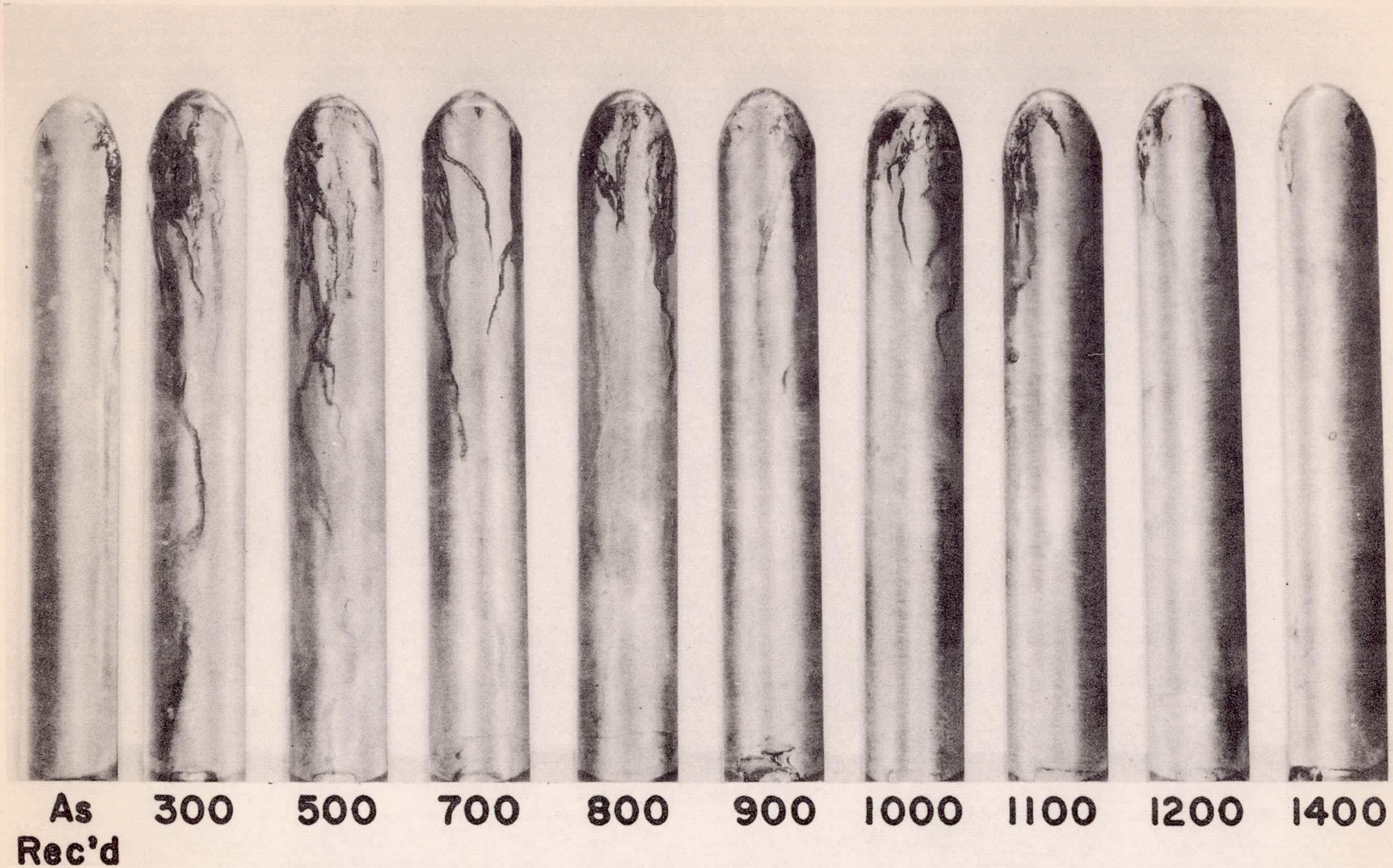
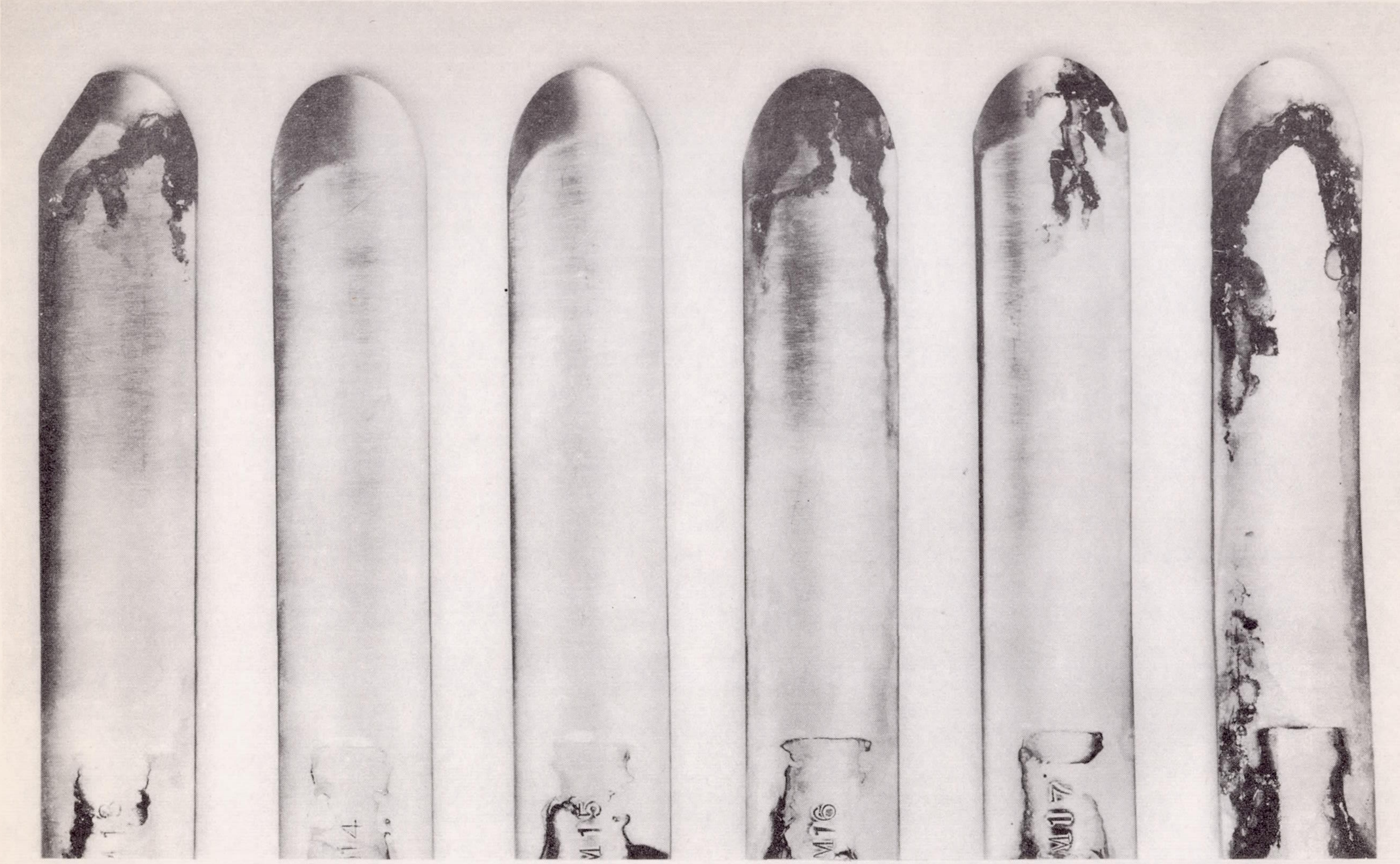


Figure 13.- 18-8-Cb (Material K) - Heat-treated specimens after 1200 hours in salt spray (20 percent). Heating period, 30 minutes. x 7/8



As Rec'd 700 900 1000 1100 1200

Figure 14.- 18-8-Mo (Material M) - Heat-treated specimens after 1000 hours in salt spray (20 percent). Heating period, 30 minutes. x 1

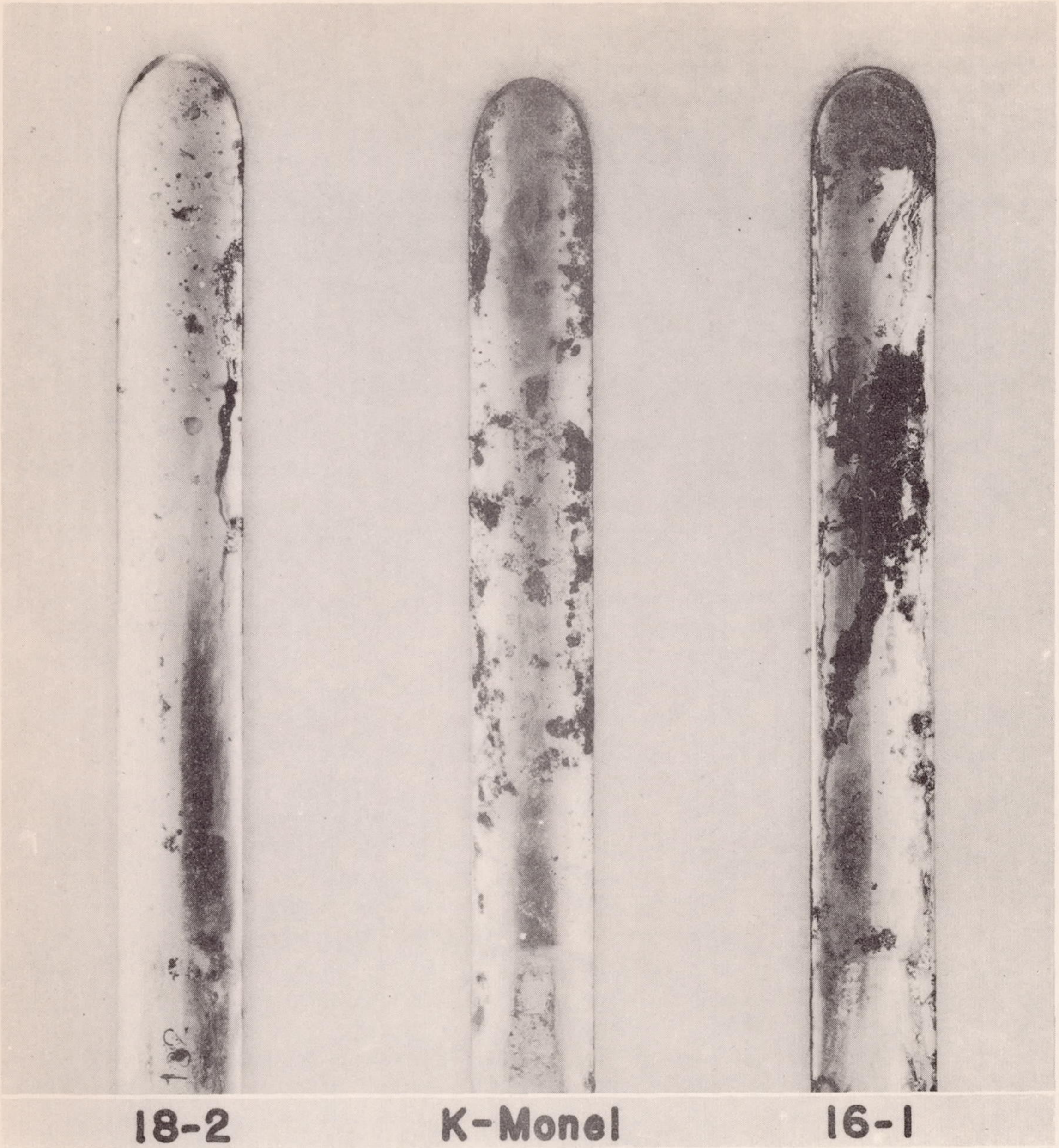


Figure 15.- 18-2, K-Monel and 16-1 tie-rod specimens after 300 hours exposure to salt spray (20 percent) in the as-received condition. Materials O, U and R. x 1

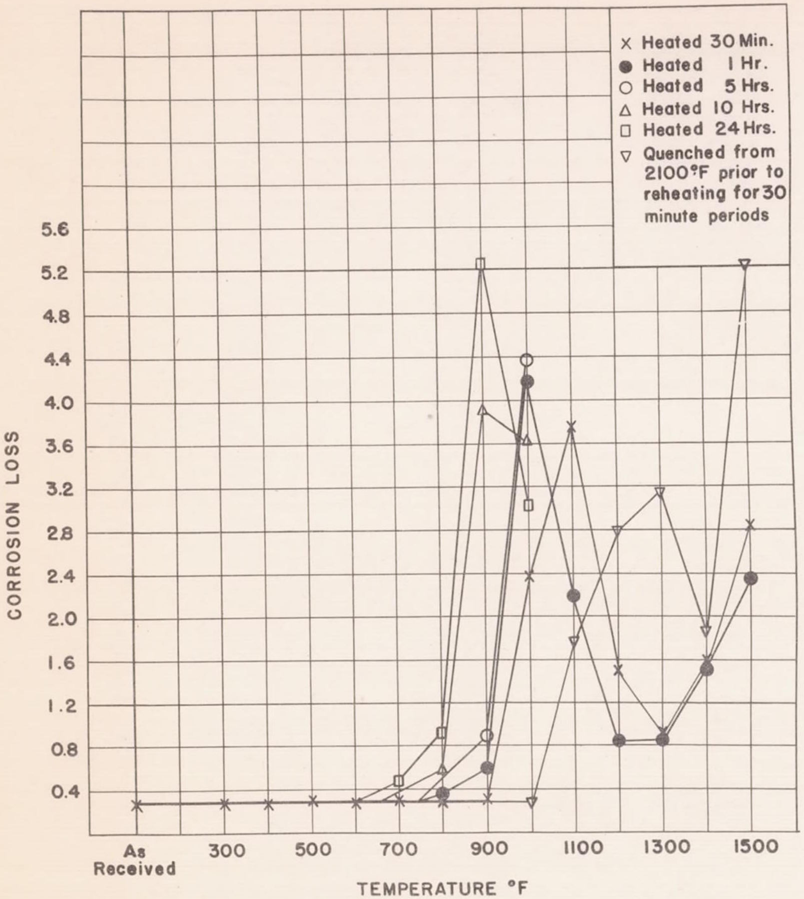


Figure 16.- 18-8 (Material E) - Percentage weight loss in boiling nitric acid (144 hours).

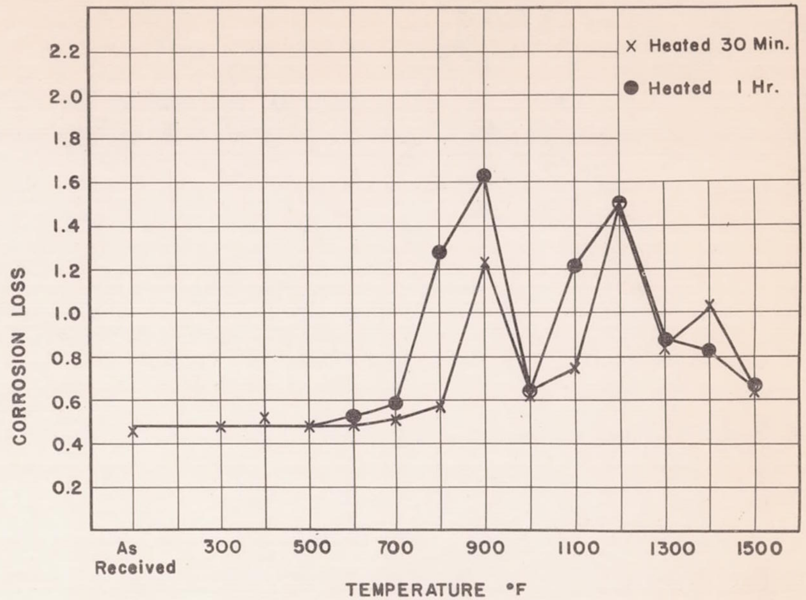


Figure 17.- 18-8-Ti (Material H) - Percentage weight loss in boiling nitric acid (144 hours).

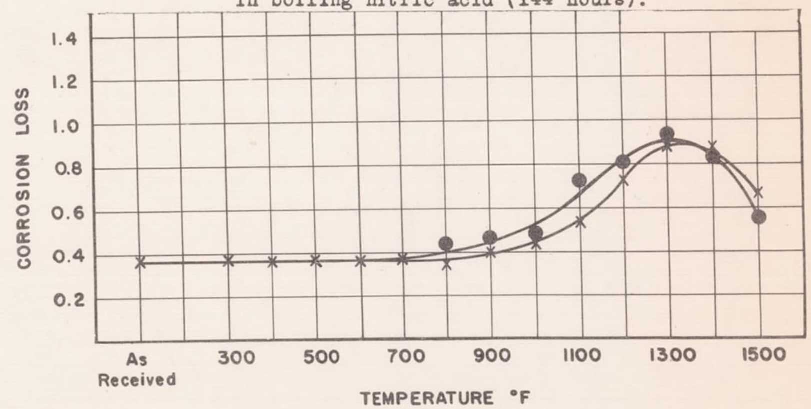


Figure 18.- 18-8-Cb (Material K) - Percentage weight loss in boiling nitric acid (144 hours).

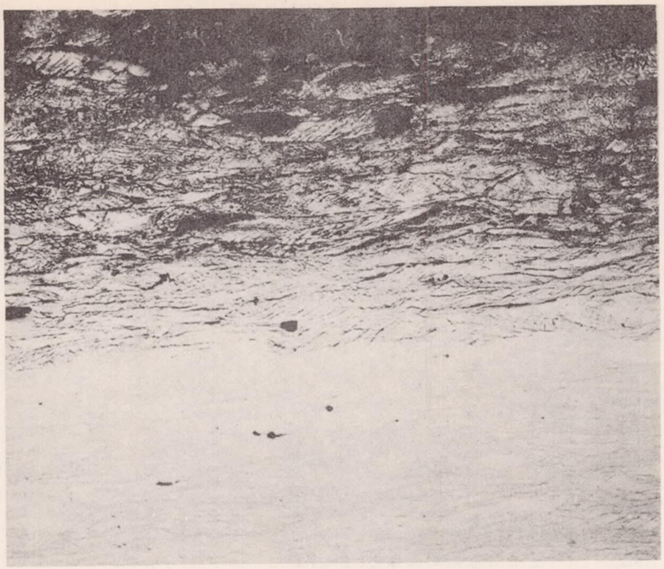


Figure 19.- 18-8 (Material E) - Type of corrosive attack produced by 100 hours exposure in boiling copper sulphate-sulphuric acid solution. Heated 30 minutes at 1100°F. Electrolytic etch in 10 percent oxalic acid. x 100

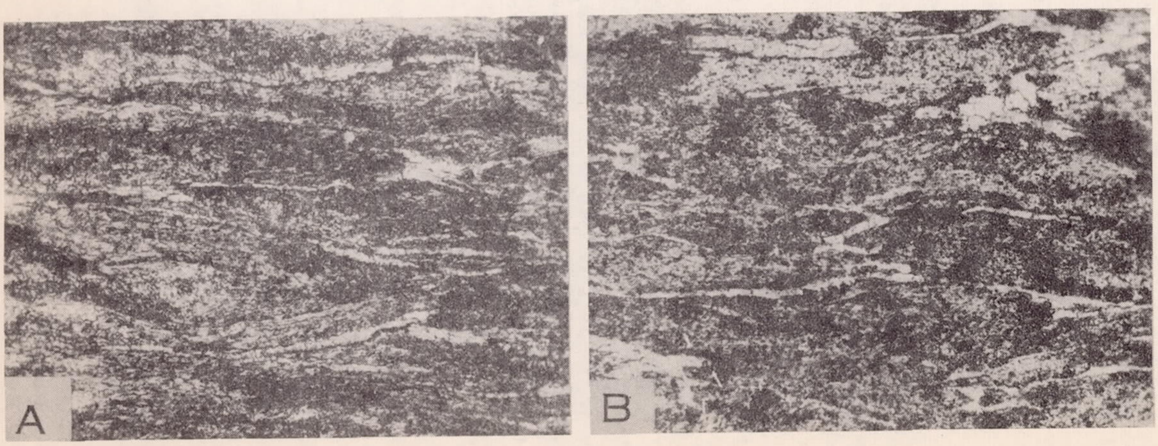


Figure 20.- 1050 steel (Material B) - A, microstructure, as received; B, heated 30 minutes at 1000°F. Etched in 1 percent Nital. x 500

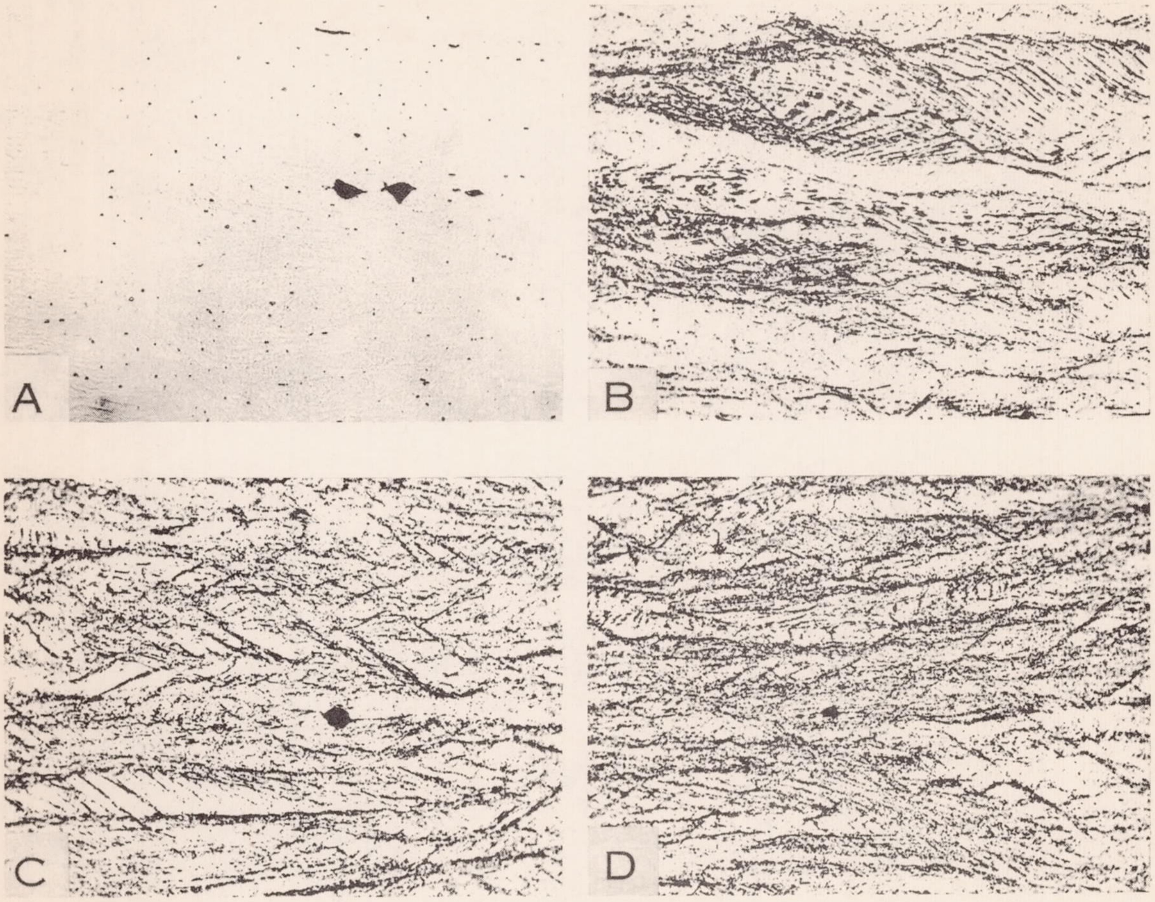


Figure 21.- 18-8 (Material E) - Microstructure of specimens heated 30 minutes at: A, 900°F; B, 1000°F; C, 1100°F; D, 1200°F . Electrolytic etch in 10 percent oxalic acid, x 500

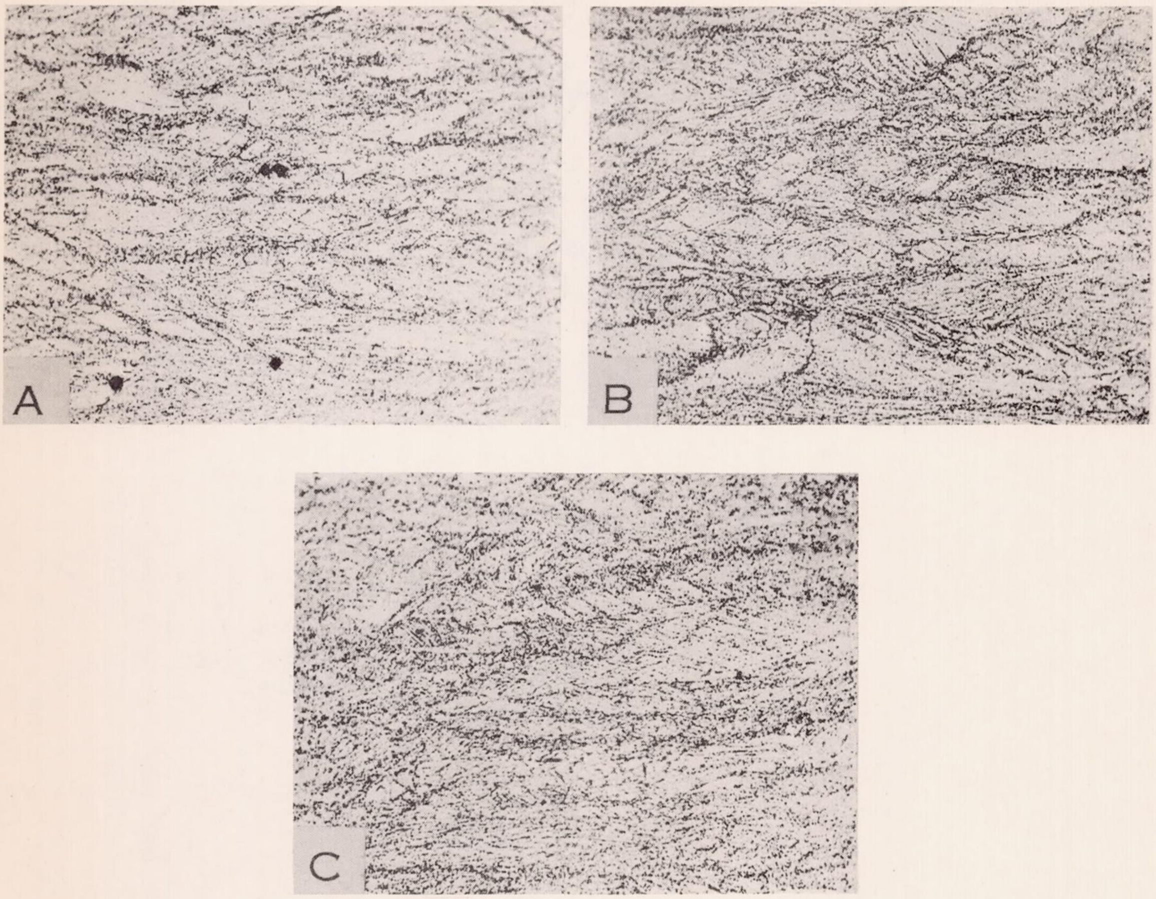


Figure 22.- 18-8 (Material E) - Microstructure of specimens heated 30 minutes at: A, 1300°F; B, 1400°F; C, 1500°F; Etched same as fig. 20. x 500

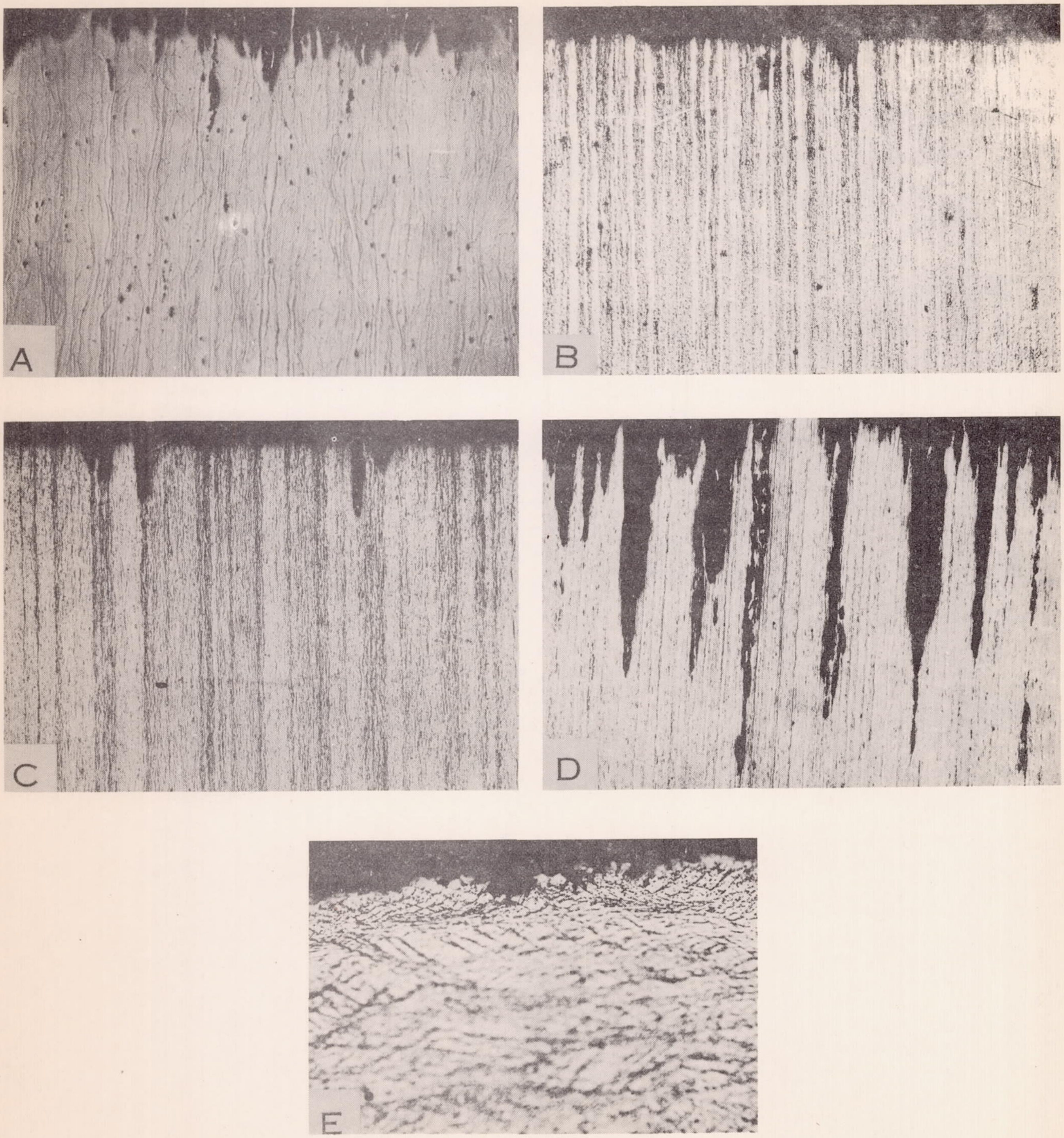


Figure 23.- Corrosive attack in boiling nitric acid;
A, End of 18-8 specimen (Material E), heated 30 minutes at 1000°F. x 100
B, End of 18-8-Ti specimen (Material H), heated 30 minutes at 1300°F. x 100
C, End of 18-8-Cb specimen (Material K), heated 30 minutes at 1200°F. x 100
D, End of 18-8-Mo specimen (Material M), heated 30 minutes at 1100°F. x 100
E, Side of 18-8 specimen (Material E), heated 30 minutes at 1100°F. x 500

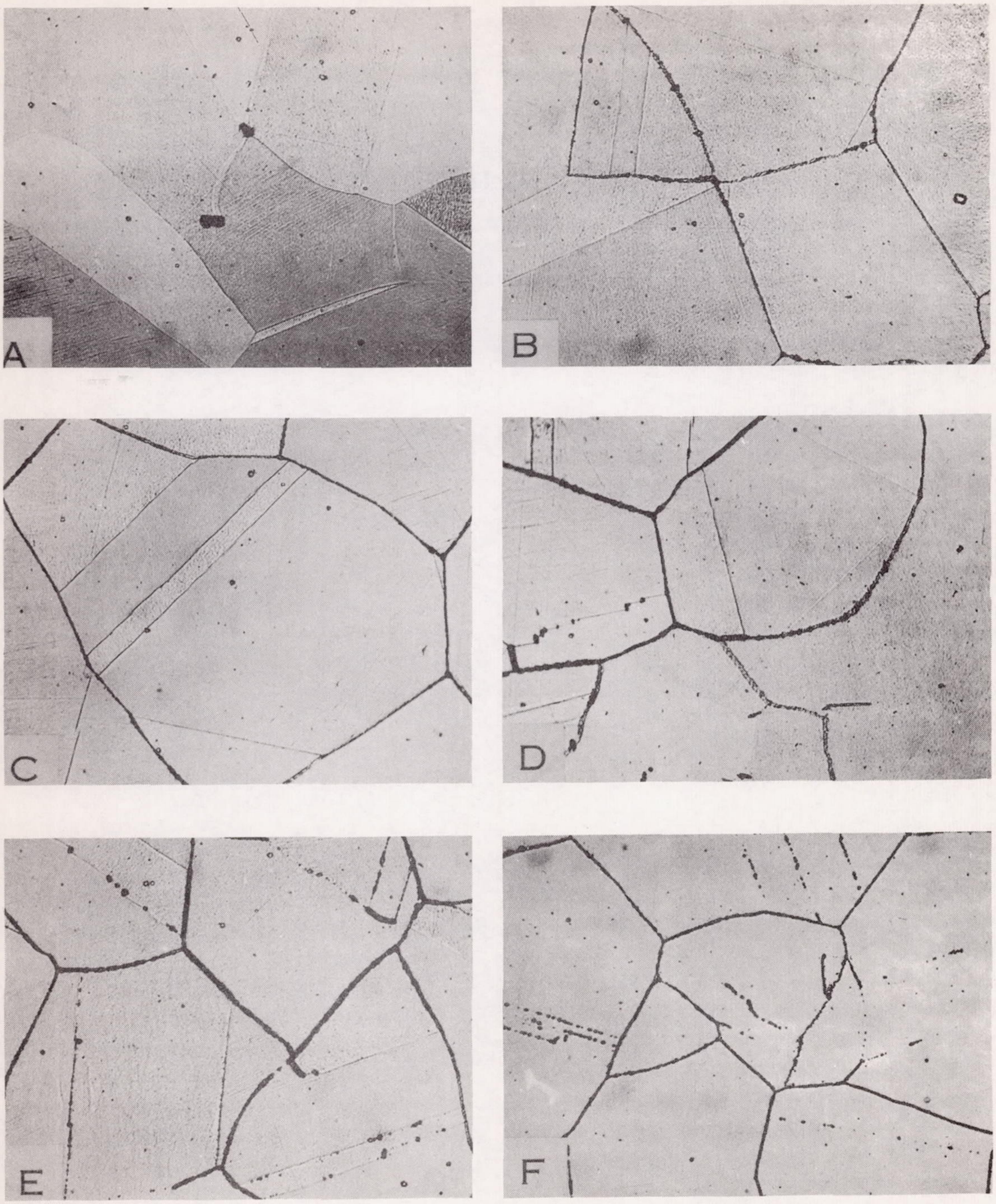


Figure 24.- 18-8 (Material E) - Microstructure of specimens quenched from 2100°F prior to reheating 30 minutes at: A, 1000°F; B, 1100°F; C, 1200°F; D, 1300°F; E, 1400°F; F, 1500°F; Electrolytic etch in 10 percent oxalic acid. x 500

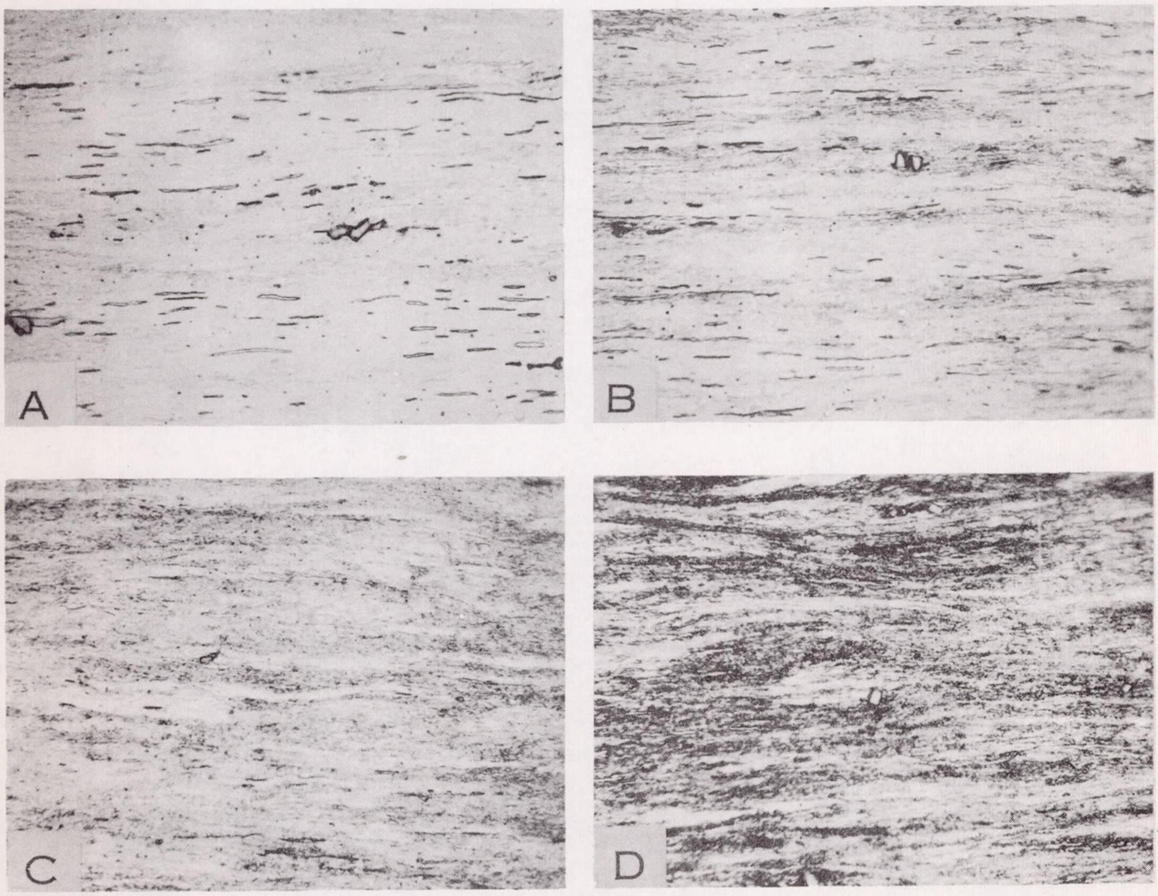


Figure 25.- 18-8-Ti (Material H) - Microstructure of specimens heated 30 minutes at: A, 800°F; B, 900°F; C, 1000°F; D, 1100°F; Etched with Vilella's reagent. x 500

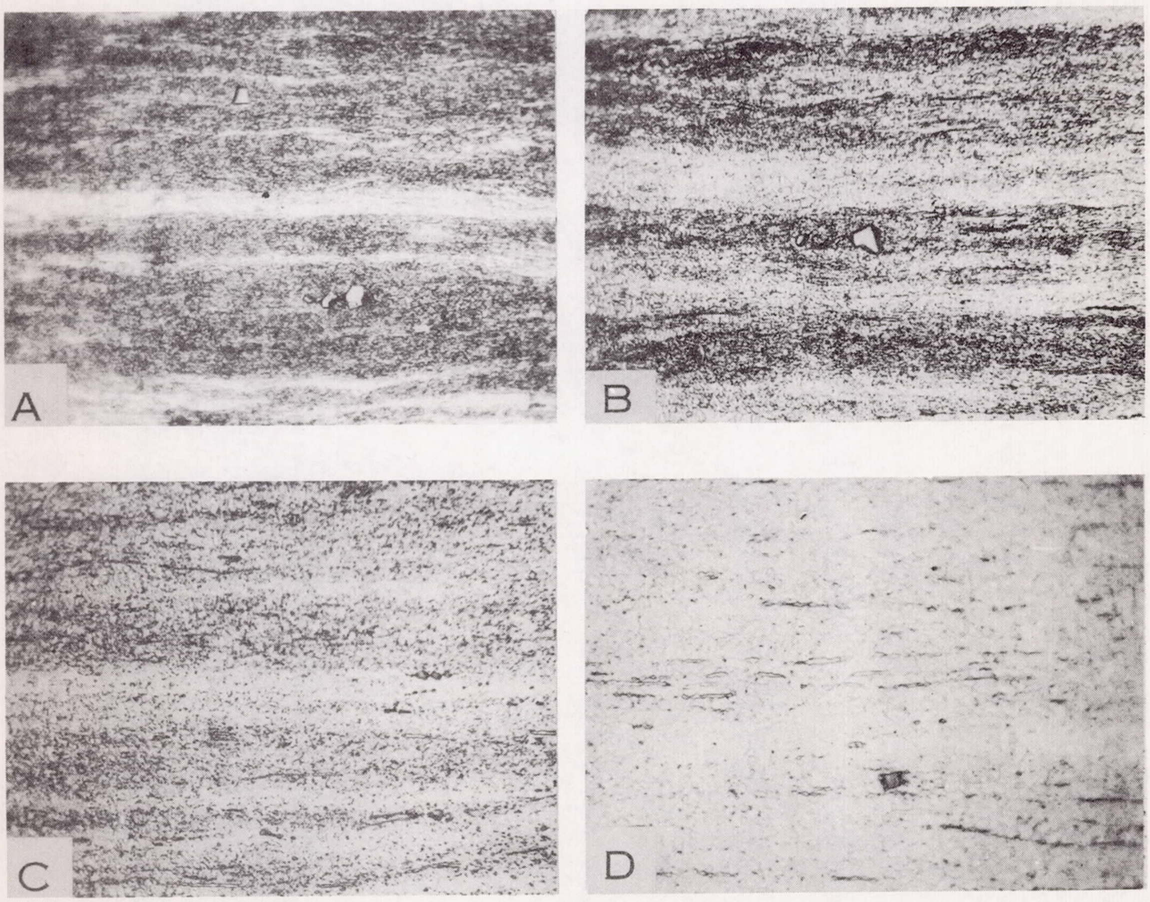


Figure 26.- 18-8-Ti (Material H) - Microstructure of specimens heated 30 minutes at; A, 1200°F; B, 1300°F; C, 1400°F; D, 1500°F. Etched with Vilella's reagent. x 500

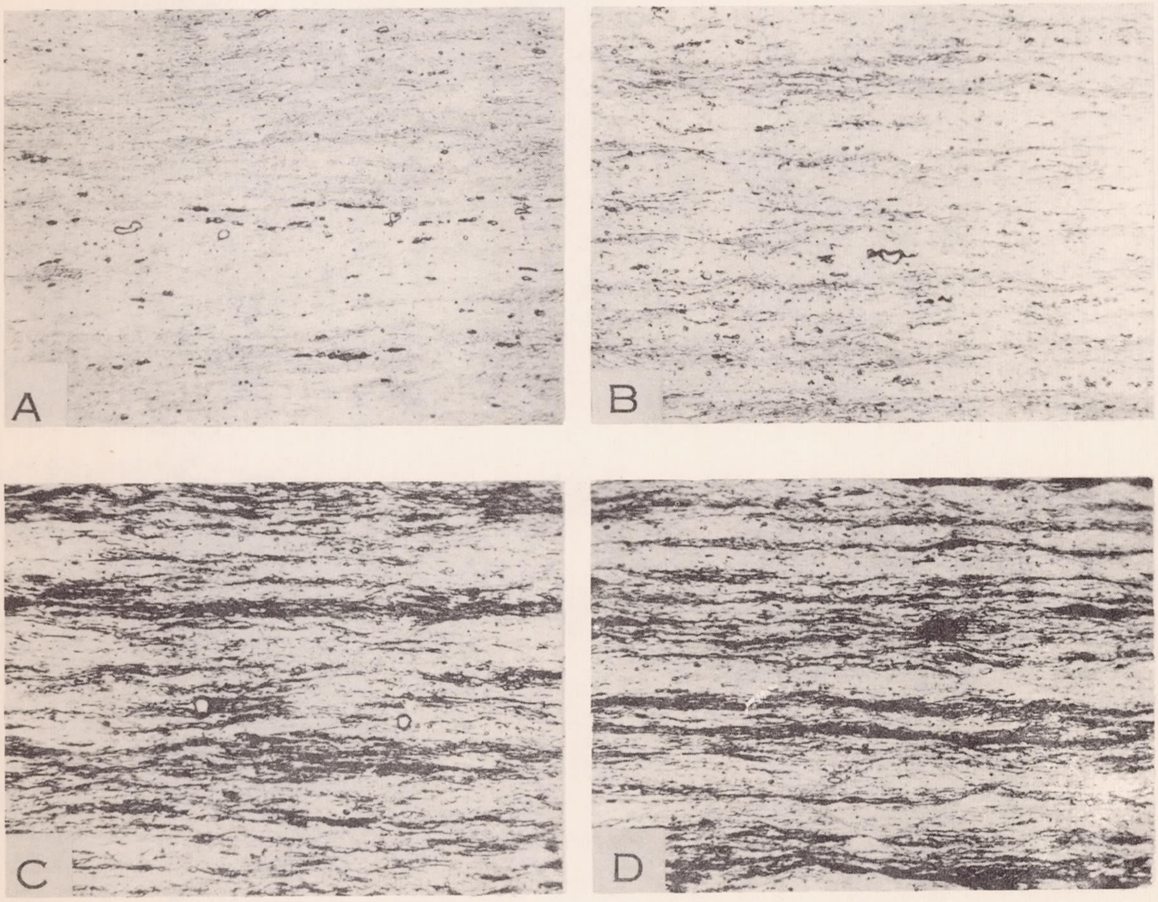


Figure 27.- 18-8-Cb (Material K) - Microstructure of specimens heated 30 minutes at; A, 800°F; B, 900°F; C, 1000°F; D, 1100°F. Etched with Vilella's reagent. x 500

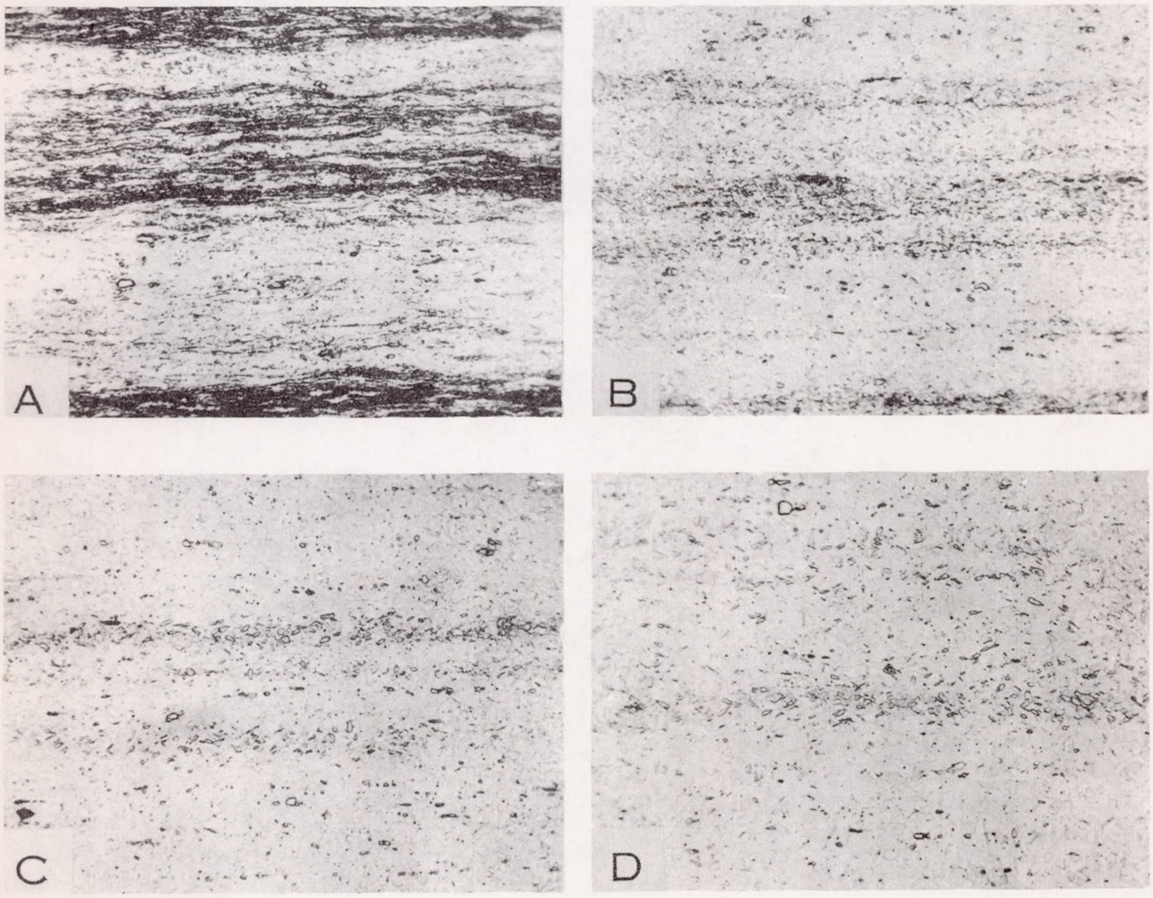


Figure 28.- 18-8-Cb (Material K) - Microstructure of specimens heated 30 minutes at; A, 1200°F; B, 1300°F; C, 1400°F; D, 1500°F. Etched with Vilella's reagent. x 500

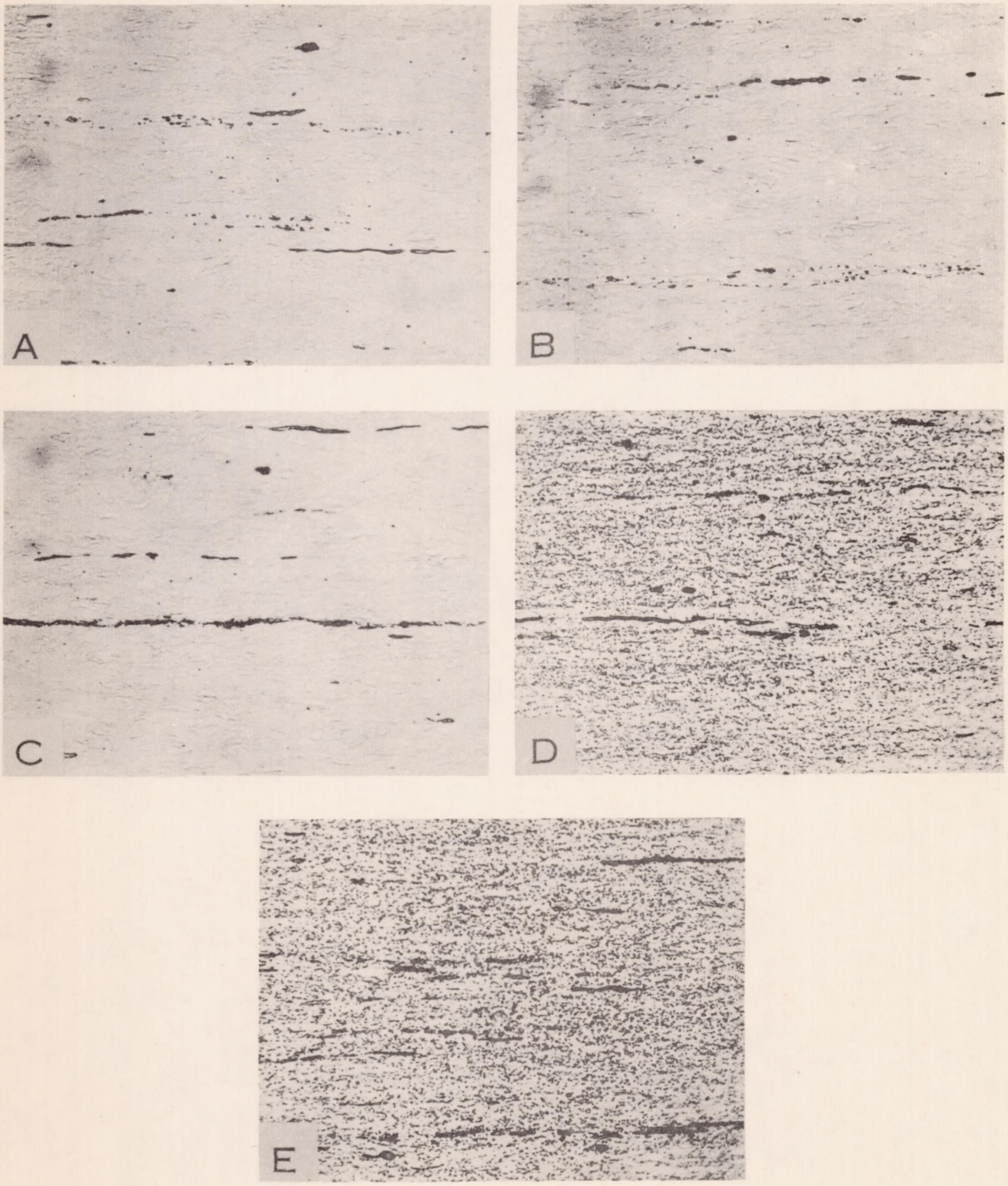


Figure 29.- 18-8-Mo (Material M) - Microstructure of specimens heated 30 minutes at; A, 900°F; B, 1000°F; C, 1100°F; D, 1200°F; E, 1300°F. Electrolytic etch in 10 percent oxalic acid, x 500

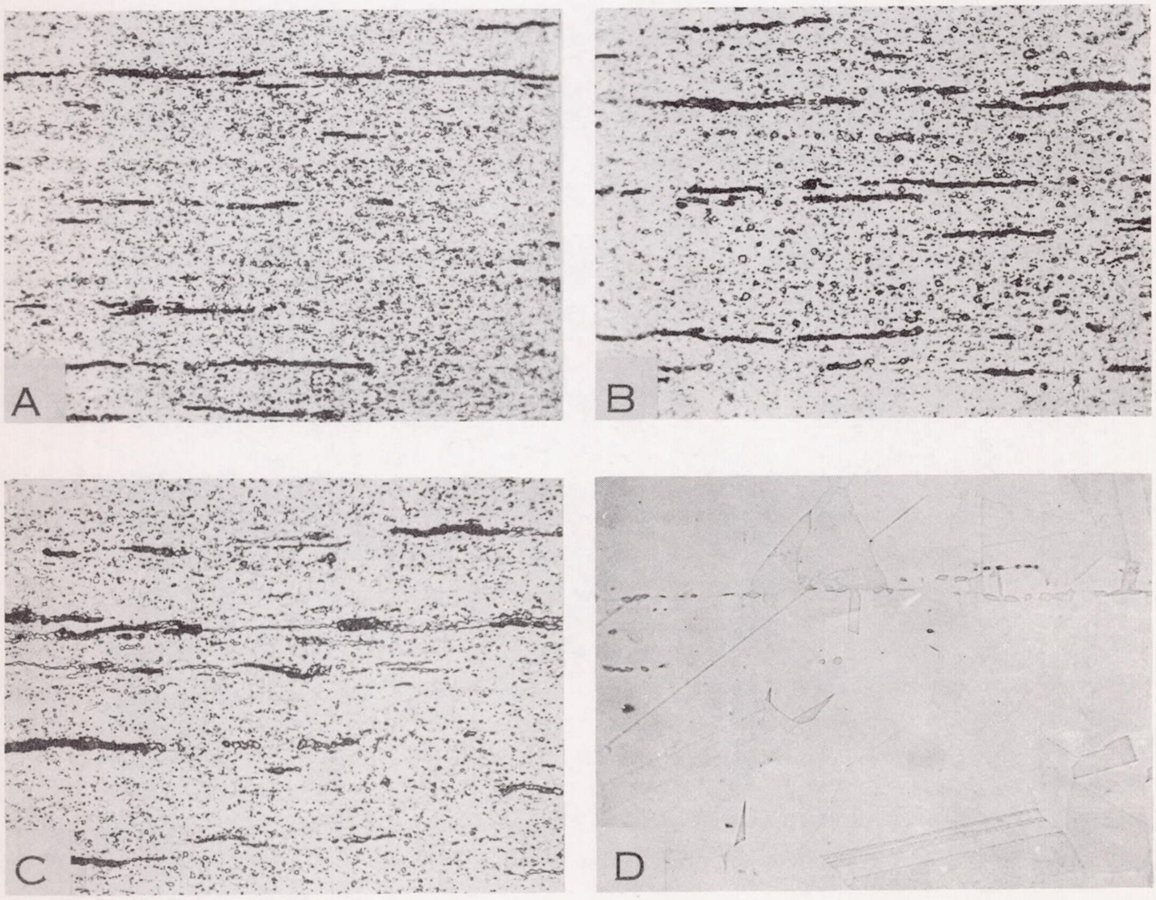


Figure 30.- 18-8-Mo (Material M) - Microstructure of specimens heated 30 minutes at; A, 1400°F; B, 1500°F; C, 1600°F; D, 1800°F. Electrolytic etch in 10 percent oxalic acid, x 500

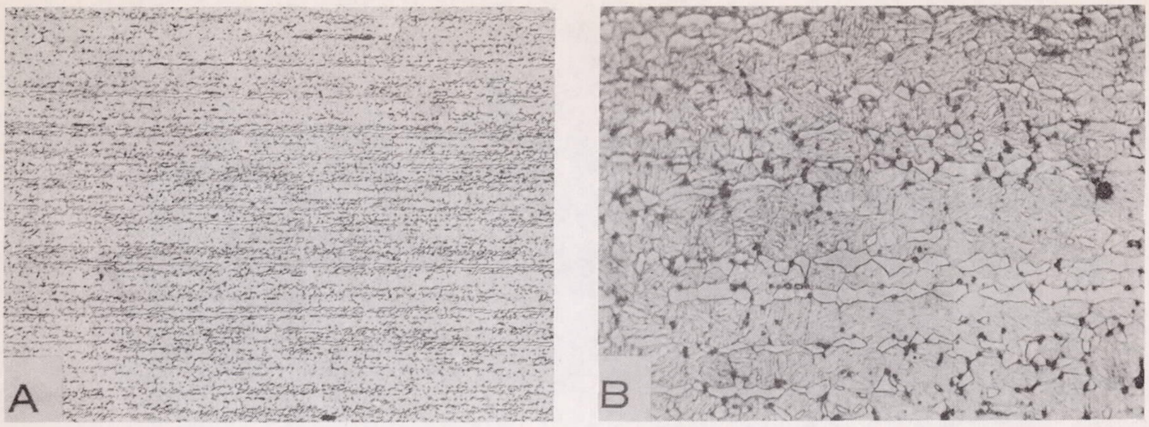


Figure 31.- 18 C_r - 2 N_i (Material O) - Microstructure in as-received condition. Electrolytic etch in 10 percent oxalic acid. A, x100; B, x 500.

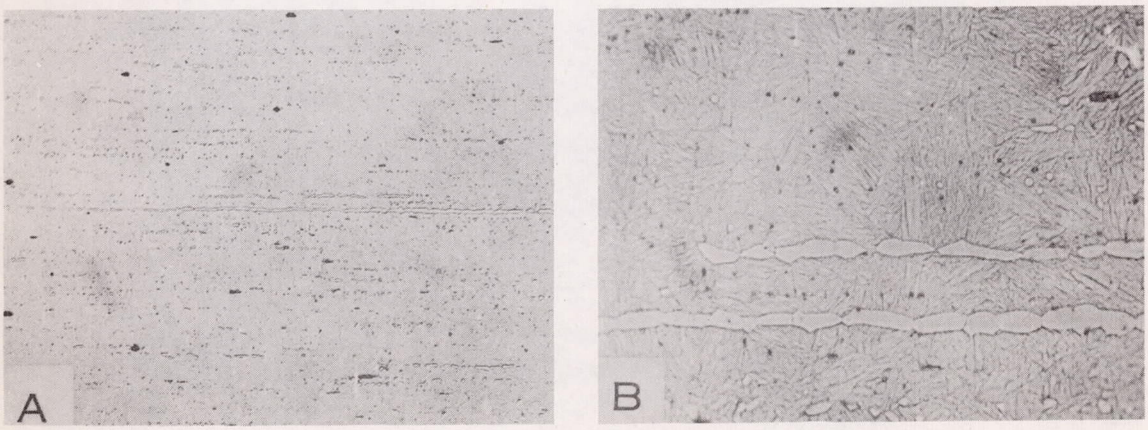


Figure 32.- 16 C_r - 1 N_i (Material R) - Microstructure in as-received condition. Electrolytic etch in 10 percent oxalic acid. A, x 100; B, x 500.

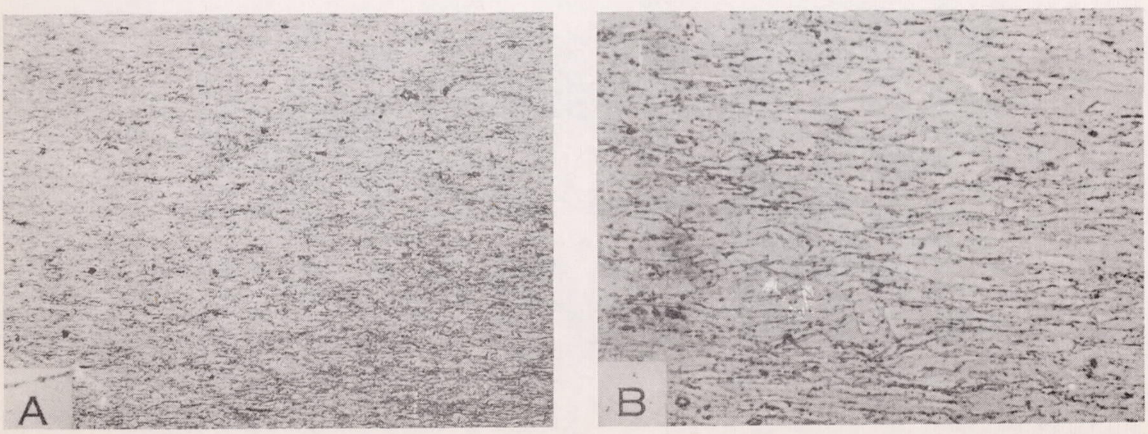


Figure 33.- K-Monel (Material U) - Microstructure in as-received condition. Etched with a mixture of acetone, acetic and nitric acids. A, x 100; B, x 500.

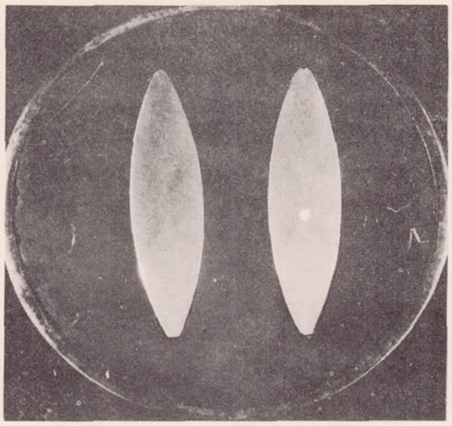


Figure 34.- X-bands in transverse sections of 18-8 tie-rods.
Electrolytic etch in 10 percent oxalic acid. x 2

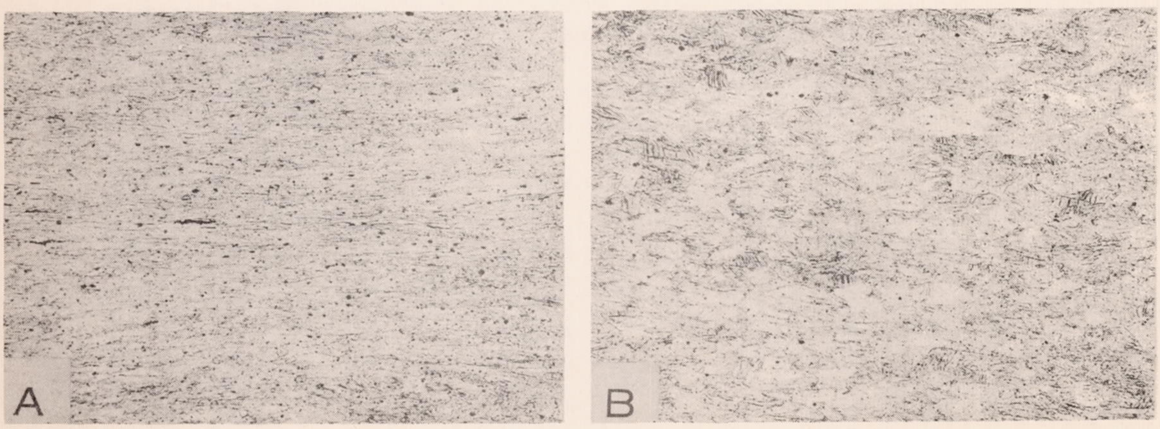


Figure 35.- A, Microstructure within X-band.
B, Microstructure outside X-band.
Electrolytic etch in 10 percent oxalic acid. x 100